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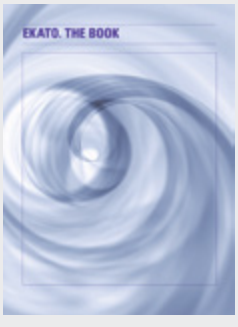

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Ad Index

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Prevent Problems with Fine Particles

Sampling is crucial and demands particular care

By Tom Blackwood, Contributing Editor

ONE OF the biggest problems encountered in solids processing is sampling particulate solids containing fine particles. Individual particles may differ in chemical composition as well as in physical size and shape. While many vendors offer equipment to address these issues, somehow designers of a process often ignore or forget about this aspect of process control.

There are legitimate reasons to minimize sample ports: contamination concerns and, maybe, cost. Sometimes, no sample is needed because of downstream processing. However, when sampling is necessary, obtaining a representative sample can be a problem for materials with wide particle-size distributions. The quantity required for a representative sample may become unreasonable. In addition, after taking a sample, subsequent handling and processing can compromise the sample integrity.

THIEF SAMPLER TROUBLE

One of the most-common ways to sample is via a thief sampler. The use of this device generally is required because sampling methods weren't built into the process design.

Unfortunately, it also is the most-abused sampling method, especially when materials contain fine particles. In such cases, it frequently gives the wrong results. For instance, we used to go to great lengths to maintain the correct particle-size distribution during manufacture. Nevertheless, a customer using a thief sampler on material in a newly arrived bulk delivery truck rejected the shipment.

Part of the problem was the way we loaded the truck from a silo. The other was having short delivery distances. At the end of a loading cycle, the chute was shaken to dislodge the final solids. Unfortunately, this freed a large fraction of fine particles that then dropped onto the top of the solids in the truck; so grab samples taken by the customer contained an excess of fine particles. Even though our silo was designed for mass flow, some of the fines collected on the chute due to electrostatic and cohesive forces. By placing a very small high-speed blender between the silo and truck nozzles, we could minimize the accumulation of fines — so that, even using a thief sampler, the customer got consistent results. It's interesting to note that trucks traveling longer distances never had this problem due to the sifting that took place during transport. We should have anticipated that the shorter distance would exacerbate the problem.

BLENDER BLUNDER

Feeding a process with multiple ingredients that widely differ in particle-size distribution presents a similar set of problems. How do you blend ⅛-in. particles with 100-µ ones — or should you even try? Unless you plan to coat the large particles, don't try!

An extrusion process in which seven chemicals were used to produce a pellet product exemplifies the problems that can arise. Loss-in-weight feeders provided the components to the extruder in the correct ratio. The process also included a blender — because during process development one had been used to mix the feed to the extruder in batch mode. The design team kept this blender in the flowsheet partly to provide a sampling point. In all of the batch studies, the blender effectively fluidized the mixture and emptied completely. The designers thought, "a blender is a blender," and chose a different type for the continuous process. Unfortunately, fines accumulated along the shell of the blender and occasionally would slough off, upsetting the composition out of the extruder. Removing the blender restored a consistent extruder product. Sometimes, simpler is better — and, yes, there are significant differences in blender design.

These examples show how complex the handling of fine solids can be, especially if you don't follow the particles' path and behavior.

- In the first case, the supplier of the product should have considered the short transport distance along with the tendency of the product to have a trailing dust cloud. Many integrating samplers that could have provided a composite sample to be sent with the truck are available; using one would have eliminated the need for a thief sample at the customer.
- The second case shows one of the hazards of converting a process from batch to continuous, and demonstrates that you have to trust your process design (i.e., the loss-in-weight feeders) to provide the correct mix.

Always remember that fine particles have a tendency not to follow the path of the other particles and to be more susceptible to segregation.

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Quickly Estimate Reagent Addition Time

A simple equation suffices in many situations involving batch reactors

By Michael J. Gentilcore, Mallinckrodt Pharmaceutical, and Luigi Grippa, Libero Professionista

BATCHES OFTEN have a step in which a reagent chemical is added to a mixture being stirred and reacts immediately (with little accumulation) — with the rate of addition controlled by the ability to remove heat. A maximum temperature is specified with full cooling applied to the reactor's jacket. If the reagent chemical is dilute enough to cause a significant level change, then the wetted area for heat transfer will not remain constant.

When the reaction rate is much faster than the feed rate, the added reagent is immediately converted to product by spontaneous reaction with the substrate previously charged to the reactor. A negligible buildup of the reagent occurs in the reactor during the addition. The heat production rate is directly proportional to the feed rate of the reagent as limited by the heat transfer of the jacket. The reagent feed rate can be raised as the volume increases and provides more wetted area for heat transfer.

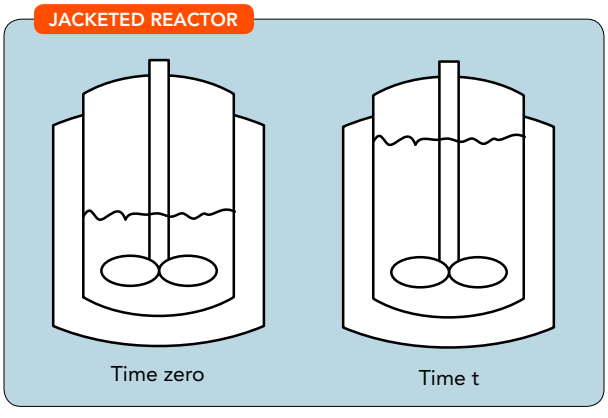

Figure 1. Equation assumes that batch always is on the straight wall of vessel.

DERIVING THE EQUATION

For simplification, let's assume the liquid mixture in the reactor prior to addition is on the straight wall as shown in Figure 1. For purposes of integration, let's also assume the heat of reaction (ΔH), liquid density (ρ), overall heat transfer coefficient (U) and temperature differential (ΔT) between the utility (jacket) and process (tank) remain constant.

This situation is the reverse of a batch distillation — for which the equations and their derivation have been published previously [1]. The solution is:

$$A_t/A_0 = e^{-t/\Theta} \quad (1)$$

where $\Theta = \rho \times D \times \Delta H/(4U \times \Delta T)$ (2)

The differences versus batch distillation are in the definition of the terms ρ, ΔH and ΔT. Let's discuss each of these.

Liquid density. In batch distillation, ρ is the density of the liquid in the vessel. For reagent addition, this term is the net weight added during the addition versus the observed volume change of the reaction mix. For the special case where the reagent and reaction mixture mix ideally with a zero volume change, the density equals that of the reagent.

Enthalpy change. In batch distillation, ΔH is the heat of vaporization of the evaporated solvent. For reagent addition, it is the heat of reaction expressed as unit of heat versus the net weight added. The heat of reaction must be calculated at the temperature of reaction and must include all enthalpy effects such as the sensible heat from a reagent below reaction temperature, heat of dilution, evaporative cooling when there is a byproduct off-gas and, of course, the chemical heat of reaction.

Temperature difference. The ΔT term is the same and constant for both batch distillation and reagent addition. Typically in batch distillation, steam is the heating medium

HEAT AND MATERIAL BALANCE

	Before Reaction		After Reaction			
	Liquid to be added	Liquid in reactor	Offgases		Reactor Contents	
	Sodium	Water	No. 1 (H_2)	No. 2 (Water)	Solute (NaOH)	Solvent (Water)
Weight, lb	1,683.5	6,610.3	73.8	14.2	2,928.9	5,276.9
Moles, lb-moles	73.2	366.9	36.6	0.8	73.2	292.9
Volume, gal	219.9	795.9			712.0	
T, °F	260.3	77.0	77.0	77.0	77.0	77.0
ΔH_f @ T, kcal/mole	1.357	-68.315	0.00	-57.796	-110.219	-68.315
ΔH_f @ T, BTU/lb	106.3	-6,831.7	0.0	-5,579.8	-4,964.6	-6,831.7
Enthalpy, kBTU	179.0	-45,159.4	0.0	-82.0	-14,540.7	-36,050.0
Density, lb/gal	7.66	8.31			11.53	
MW, lb/lb-mole	22.9898	18.01528	2.0158	18.01528	39.997	18.01528

Table 1. Sodium added to vessel causes a reaction.

NOMENCLATURE

- A area, ft^2, m^2
- Cp Coolant heat capacity, BTU/(lb×°F), J/(Kg×-°C)
- D Tank i nside diameter, ft, m
- ΔH Heat of reaction, BTU/lb, J/kg
- K Flow rate correction per Eq. 4, dimensionless
- t Temperature, °F, °C
- ΔT Temperature difference (jacket - process), °F, °C
- T Time, hr
- U Overall heat transfer coefficient, BTU/(hr×ft^2×°F), W/(m^2×-°C)
- W Coolant flow rate, lb/hr, kg/s
- ρ Liquid density, lb/ft^3, kg/m^3
- Θ Time constant per Eq. 2, hr

Subscripts

- 0 Time zero
- f Formation
- t Time t
- S Coolant supply (jacket inlet temperature)
- O Coolant return temperature (jacket outlet temperature)
- P Process temperature (reaction temperature)

and calculation of this term is straightforward because both the process and jacket are isothermal. In reagent addition, a liquid commonly is the coolant and the jacket supply and outlet temperature are unequal. In this case, the difference is determined by a log mean temperature calculation.

For a simple case of water flowing once through a jacket, the following relationships apply:

$$T_O = T_P + (T_S - T_P)/K \quad (3)$$

where $K = \exp[(UA)/(WCp)]$ (4)

Eq. 4 includes the wetted area (A). Under a rigorous analysis, the temperature difference is not constant. However, as an approximation, the log mean temperature difference can be calculated at both the lowest level (A_0) and highest level (A_t) in the integration. For practical problems, the observed disparity in temperature difference will be slight and the lower value can be used to provide a conservative estimate of addition time. An example will illustrate this.

Many reactors have heat/cool modules that enable the jacket inlet temperature to differ from the coolant supply temperature. In such cases, substitute equations that are available in Reference 2 for Eq. 3 and Eq. 4.

AN EXAMPLE

Molten sodium metal is added to water to make a sodium hydroxide solution — with the temperature controlled isothermally at 77°F during the addition. The reaction chemistry is:

$$Na\ (liq.) + H_2O\ (liq.) \rightarrow NaOH\ (aq.) + \tfrac{1}{2}H_2\ (gas)$$

The reaction occurs in a vessel with a 5-ft outer diameter and a ¼-in.-thick shell, equipped with a bottom ASME F&D head. The straight wall holds 144.5 gal/ft of liquid height. The bottom head holds 73.9 gal. The straight side has a wetted area of 15.7 ft^2/ft of liquid height. The outside surface area

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"Quickly Estimate Batch Distillation Time," http://goo.gl/3je2cI

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REFERENCES

1. Gentilcore, M. J., "Quickly Estimate Batch Distillation Time," *Chemical Processing* (April, 2013), http://goo.gl/3je2cI.
2. Gentilcore, M. J., "Estimating Heating and Cooling Times for Batch Reactors," *Chemical Engineering Progress* (March 2000).

of the bottom head is 23.2 ft^2. Table 1 presents a heat and material balance for this reaction.

Let's now estimate the addition time, assuming an overall heat transfer coefficient of 80 BTU/hr/ft^2/°F and 25,000 lb/hr of 41°F chilled water flow (Cp = 1) to the jacket.

Step 1. Calculate the liquid density, ρ.

Per Table 1, the change in volume is 712.0 - 795.9 = -83.9 gal = -11.2 ft^3. The change in weight is 2,928.9 + 5,276.9 - 6,610.3 = 1,595.5 lb. The liquid density for this reaction is 1,595.5 lb/(-11.2 ft^3) = -142.5 lb/ft^3. (Surprise! The density is a negative number because the total volume shrinks after the addition.)

Step 2. Calculate the heat of reaction.

Per Table 1, the heat liberated is the sum of the product enthalpies minus the sum of the reactant enthalpies, i.e., [0.0 + (-82.0) + (-14,540.7) + (-36,050.0)] - [179.0 + (-45,159.4)] = -5,692.3 kBTU. The net weight added to the reactor is 1,595.5 lb. (Surprise! It doesn't equal the weight of the sodium metal because of the evolution of hydrogen gas.) The heat of reaction is then -5,692.3 kBTU/1,595.5 lb = -3.568 kBTU/lb or -3,568 BTU/lb.

Step 3. Calculate the height on the straight wall.

Start of reaction: (795.9 - 73.9)/144.5 = 5.00 ft

End of reaction: (712.0 - 73.9)/144.5 = 4.42 ft

Step 4. Calculate wetted areas.

Start of reaction: 5.00×15.7 + 23.2 = 101.7 ft^2

End of reaction: 4.42×15.7 + 23.2 = 92.6 ft^2

Step 5. Calculate K factors. (See Eq. 4.)

Start of reaction: K = exp (80×101.7)/(25,000×1) = 1.385

End of reaction: K = exp (80×92.6)/(25,000×1) = 1.345

Step 6. Calculate jacket outlet temperatures. (See Eq 3.)

Start of reaction: T_O = 77 + (41 - 77)/1.385 = 51.0°F

End of reaction: T_O = 77 + (41 - 77)/1.345 = 50.2 °F

Step 7. Calculate ΔT.

Start of reaction: [(77 - 41) - (77 - 51.0)]/ln [(77 - 41)/(77 - 51.0)] = 30.7°F

End of reaction: [(77 - 41) - (77 - 50.2)]/ln [(77 - 41)/(77 - 50.2)] = 31.2°F

The two values differ by less than 2%. Per the integration assumptions, the temperature will be treated as constant — using the lower value, 30.7°F, in subsequent calculations to give a conservative estimate of the addition time.

Step 8. Calculate Φ. (See Eq 2.)

First, calculate the tank's inside diameter:

5 - 2×¼×½ = 4.9583 ft

Φ = [(-142.5)×4.9583×(-3,568)]/(4×80×30.7) = 256.6 hr

Step 9. Rearrange Eq. 1 to solve for time.

$t = -\Phi \ln(A_t/A_0)$

Step 10. Plug in values and calculate addition time.

-256.6×ln(92.6/101.7) = 24.1 hr

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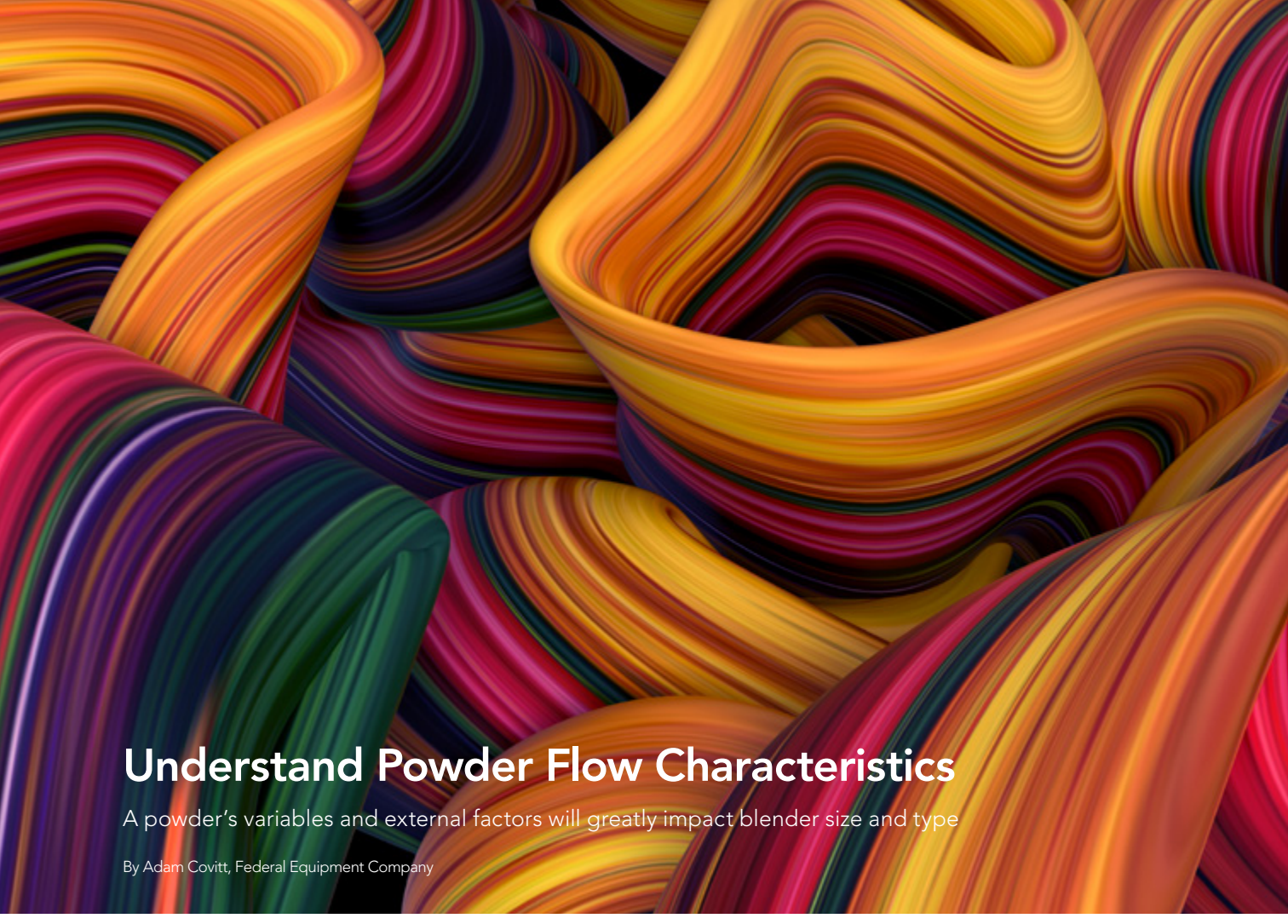

Understand Powder Flow Characteristics

A powder's variables and external factors will greatly impact blender size and type

By Adam Covitt, Federal Equipment Company

THE GOAL of powder blending is a homogeneous, or uniform, consistent mix of materials. The key to selecting a blender that will blend powders together into uniform mixes, also called "bulk solids," is the material's flow characteristics. Free-flowing powders are more likely to blend well together, but not all powders are free-flowing. Understanding the powder's flow characteristics will help determine the best type of blender for the product.

POWDER FLOW CHARACTERISTICS

Powders are the least predictable of all materials in terms of their ability to flow. Two sets of factors determine a powder's flow characteristics: the powder's variables and external factors. Powder variables include the product's bulk density, particle size, size distribution, shape, surface texture, cohesiveness, surface coating, and electro-static charge, among others. External factors include vibration, temperature, humidity, spurious electrical charges, aeration, container surface effects (or wall friction) and storage time. Addressing only one set of variables or partially addressing both sets of variables will lead to flow and, eventually, blend uniformity problems on the production floor. The flow characteristics will help identify the proper type of blender and the powder's weight and density will help determine the size of the blender.

BLENDER SIZE

Blenders are volumetric, which means their sizes are usually measured in terms of their volume capacity such as cubic feet. Powders will not blend well (flow) if the blender is too full or too empty. A safe range of effectiveness is 35–65% of the overall capacity. The product's flow characteristics will be the best indicator for the capacity range of each product mixed in the blender. As a rule of thumb, working capacity of a blender is usually determined as 50% of the total volume of the blender. For example, if a blender has an actual volume of 2 ft^3, then the working capacity will be 1 ft^3.

While blenders are generally sized according to volume, powders are usually measured according to density. Such measurements are either grams per cubic centimeter (gr/cc) or pounds per cubic foot (lbs/ ft^3). Additionally, the request for blended materials usually comes in terms of a specific weight of material such as 15 kilograms (kg) without reference to volume or density. Powders can be

very light ("fluffy") or very dense which leads to different volumes of product at the same weight (think of a pound of feathers versus a pound of lead).

The proper blender size for the product is one that will have a working capacity within the effective range to achieve a uniform blend. The simple way to calculate the proper size versus the product density is to weigh a quart or liter of powder. 16 quarts or 15 liters of a product is equal to 1 ft^3 at a density of 35 lbs/ ft^3. Blenders sizes based on 35lbs/ ft^3 at working capacity: 1 ft^3 = 15kg; 2 ft^3 = 30kg; 3 ft^3 = 45kg; 5 ft^3 = 75kg; 10 ft^3 = 150kg; 20 ft^3 = 300kg; 30 ft^3 = 450kg; and 50 ft^3 = 750kg. Size is not the only factor to consider, the type of blender is important as well.*

BLENDER TYPES

Twin-shell (or "V" blenders), double cone blenders and bin (or tote) blenders are all considered "random" style blenders. These types of blenders also are referred to as "open shell blenders." They randomly mix powders that are already free flowing through the blender's tumbling action. If the products are dense, an intensifier bar can be added which will force the powders to move inside the shell of the blender. Liquids may be added to the bulk solids mixture with a liquids bar. Intensifier bars can be overused which can result in particle break down for dry and friable powders or it can pack powders that are wet or cohesive (sticky).

Ribbon and paddle blenders are excellent ways to mix powders slowly and gently. In a ribbon blender, a double-helix agitator will move materials towards the center of a trough with the outer blade while the inner blades move the materials towards the outside of the trough. Paddles are an alternate design that can be used for small batches relative to the working capacity of the blender and with friable materials.

High shear mixers are generally used for products which are considered immiscible, where the products to not generally form a homogeneous blend. The mixer operates by moving one phase into a continuous phase. The phases, or ingredients, can be solids, liquids or gases. The ingredients are moved with a rotor, or impeller, across other rotors or stators to produce a mechanical force called shear which forces the products to mix. Plow-style mixers are common high shear mixers that can be used for particle size reduction and to produce granules.

These are only a few, common-types of blenders available. Many additional types of mixers can be used for powder blending including planetary mixers, Nauta-style mixers and single and double-arm mixers — choices among many others which can be evaluated applying the same analysis of weight, capacity and flow characteristics. Powder flow characteristics and the weight of the product can be used to determine the proper type and size of the blender needed for the product. Analyzing the weight of the product can be used to estimate the working capacity required which determines the overall size of the blender needed. The flow characteristics will also determine whether the product can be blended in a random type blender, a ribbon or paddle blender, high shear mixer or some other type of blender. ●

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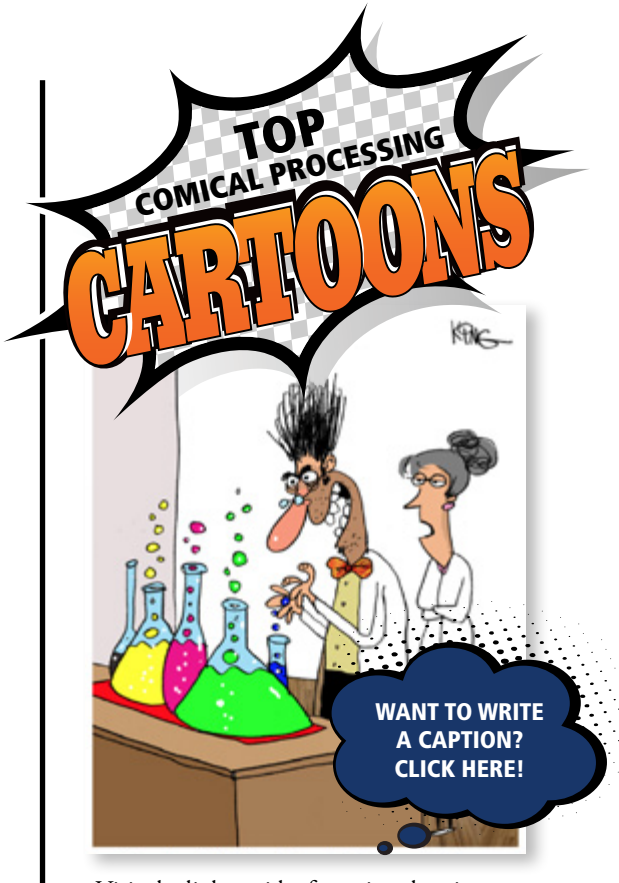

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