Tube Selection for Analyzer Sample Systems
Selecting the optimum process tube for use in analyzer sample transport systems requires critical consideration of process conditions. There are a wide variety of metallic and nonmetallic tube types available: welded and seamless 304 and 316 series stainless steel, other metallurgies used include Monel®, Hastelloy®, Inconel®, Incoloy®, zirconium, SuperDuplex, titanium, and even copper. Common nonmetallic tube materials are Teflon® (PFA, FEP, and PTFE fluoropolymer), polyethylene and Halar (ECTFE). Some bundles may also contain coaxial tubing (tube-in-tube) or unheated tubes for calibration gas and blowback lines.

O’Brien provides a wide variety of in house services including chemical passivation, thermocouple cleaning and cleaning for oxygen service. We also furnish chemically passivated tubing with an elemental silicon coating from SilcoTek.

TrueTube® is a family of tubing products developed by O’Brien to provide reliable and repeatable performance in sample transport for process and emissions analyzers. O’Brien Analytical starts with select ASTM grade tubing and enhances the physical characteristics to improve dry-down, adsorption and corrosion resistance. We do this by controlling surface roughness, cleanliness, surface chemistry and reactive contaminants.

Improper tube selection may lead to failure of the analyzer system caused by adsorption, contaminants, corrosion stress cracking or gas permeation in the sample transport line. Restricted sample flow, long sample lag times, and kinking are also problems often related to tube selection.

Cleanliness and Reactive Contaminants

The O’Brien TrueTube process removes drawing oils and other contaminants that are often present in commercial grade tubing. At the same time surface iron is removed and the tube is left with an enriched Cr/Fe and CrO/FeO ratio which resists corrosion.

Selecting Tube Material
Consider the level of protection needed against corrosion, porosity, and adsorption as well as the size, wall thickness and metallurgy (or coating) of the tubing.

Corrosion
When determining corrosion resistance it is imperative that the material selection considers failure conditions. Just because the process stream does not contain acids under normal conditions the component concentrations and byproducts created if an upset occurs must also be considered. The creation of acids is common in stack gas applications when inadequate heating allows vapor to condense and combine with sulfur or nitrogen compounds.

Porosity
Fluoropolymer tubes are common for many analyzer applications particularly stack gas measurement. For all of its positive characteristics fluoropolymer is “porous” and has a limited working pressure. Porosity and working pressure deteriorate rapidly as temperature increases. Consider using only thick wall (0.062” / 1.5mm) tubing.

If the porosity and / or pressure rating is not acceptable then consider using electropolished or SilcoNert™ 2000 silicon coated stainless steel tube. While resistant to most chemicals silicon coatings have limited resistance to acids so caution should be exercised.

Adsorption
When using stainless steel for the transport tubing, adsorption and desorption will exhibit itself as a delay in detecting changes of trace amounts of component (especially moisture and sulfur compounds, H₂S and SO₂.) The structure of commercial stainless steel tubing is such that compounds may be caught up in the “crevices” and not reach the analyzer.
Once the tube is saturated the levels will equalize relative to the process as long as the process composition, temperature and pressure remain constant. Any change in composition, temperature or pressure will cause compounds to be adsorbed at a different rate by the tube or released at different rates from the tube surface and create a false “spike” in the analyzer readings. As a result adsorption and desorption create inconsistent and unpredictable results. This is sometimes referred to as the “memory effect”. This problem can often be solved by the use of electropolished and/or SilcoNert coated tubing.

SilcoNert 2000 – This is an inert, chemically protective patented barrier of amorphous silicon material inter-diffused on our TrueTube CP chemically polished 316L substrate resulting in a 100-250 nm coating. The treated surface is further passivated with covalently bonded hydrocarbon molecules. The process is verified by an average surface contact angle value greater than 75° using deionized water. This process replaces Silcosteel and has also been known as Siltek and Sulfinert. It is used in our TrueTube FS and TrueTube EPS products.

SilcoNert 2000 is the most inert coating we have found, however it is limited by alkaline/base compounds as well as fluorides.

Dursan – Is the newest coating from SilcoTek. It is a patented abrasion resistant durable, inert, corrosion resistant carbon based coating that is harder than steel and highly resistant to acid and base attack. The intermolecular coating is bonded to our TrueTube CP chemically polished 316L substrate in a minimum 500 nm thickness. With a contact angle greater than 100° Dursan is a hydrophobic surface that strongly repels water. It is unaffected by both acid and alkaline through the full 0-14 pH.

Dursan is recommended for any high pH stream or where stream abrasion is a problem. It is used in our TrueTube HCR product.

The O’Brien chemically polished and electropolished tubing provide the ideal surfaces for bonding with either SilcoNert 2000 or Dursan coatings.

Surface Roughness
Surface roughness contributes to adsorption / desorption problems associated with analyzer sample lines. The microphotographs below illustrate one of the differences in tube material and selection. Surface roughness is only one factor to consider when specifying sample tube material. However, like other choices in sample system components, it can improve or limit the repeatable accuracy of the entire system.

Coatings
Inert coatings can be applied to the ID of sample transport tubing that dramatically change the sample system response time. This retains the pressure and temperature benefits of metallic tubing and eliminates the interaction of the base metal with a specific analyte.

We utilize two coatings from SilcoTek in our TrueTube family of sample transport tubing: SilcoNert™ 2000 and Dursan™.

Guidelines for selection of sample transport tubing:
The choice of sample transport tubing is more critical when the sample is in gas phase rather than liquid phase. As the concentration of the analyte decreases the selection of sample transport tubing has a greater influence on the system response time.

Fluoropolymer
There are several varieties of fluoropolymers; PFA, PTFE, and FEP are the most common choices for tubing. Fluoropolymers are chosen because they are essentially chemically inert. They are an ideal transport medium for highly volatile chemical compounds and exotic fluids.

There are very few chemicals, such as fluorine, chlorine trifluoride, and oxygen difluoride that are known to react with fluoropolymers. To a lesser degree, fluoropolymer tubing may absorb halogenated organic chemicals. This causes swelling and change in weight.

Permeability is a major consideration with fluoropolymer tubing. The amount of permeation depends upon several factors; the porosity of the tube material, the tube thickness, the molecular size of the permeant, and the relative concentration of the permeant inside and outside the tube. Increasing the temperature always increases the permeation rate. Increasing the wall thickness always reduces the permeation rate.

Pressure rating at operating temperature must be considered. The allowable pressure rating for fluoropolymer tubes decreases rapidly with increasing temperature. However, many analyzer applications using fluoropolymer tubing are operated at a slight vacuum.
316L Seamless Stainless Steel
Commercial grade 316L SMLS SS is used in many sample transport applications. Because of differences in manufacturing processes the surface roughness of commercial grade seamless tubing can vary widely. Commercial grade seamless tubing often has a higher surface roughness than a similar welded tube. Drawing oils and other contaminants from the manufacturing process may cause problems with the accuracy of sample analysis until the system is flushed and cleaned.

TrueTube CP
Select seamless 316L stainless steel tubing is chemically polished and cleaned to produce a surface finish with less than a 40 μ-inch surface roughness. TrueTube CP also meets the requirements of ASTM A632-S3 for thermocouple cleaning and CFOS per ASTM G93 Level A and CGA g-4.1. It contributes to improved sample transport times by reducing surface roughness and eliminating contaminants.

TrueTube FS
A secondary process creates a SilcoNert™ 2000 elemental silicon coating on the inside diameter of chemically polished and cleaned seamless 316L stainless steel tubing. The silicon coating improves the corrosion resistance and reduces the affinity of stainless steel to many compounds such as H₂S. SilcoNert 2000 coated tubes have found acceptance for transporting low concentration sulfur samples.

TrueTube HCR
A secondary process creates a Dursan™ elemental silicon/carbon coating on the inside diameter of chemically polished and cleaned seamless steel tubing. The hard scratch resistant silicon/carbon coating improves the corrosion resistance and inertness of stainless steel. It is pH stable in exposure to both acid and base solutions. While not as inert as the SilcoNert 2000 it is resistant to base compounds and exhibits excellent corrosion resistance.

TrueTube EP
The photos illustrate the superior surface finish achieved by O’Brien electropolished TrueTube EP. Electropolishing not only improves surface roughness but also provides enhanced corrosion resistance by leaving a chromium enriched surface layer. The Cr:Fe ratio will be a minimum of 1.5:1 and CrO:FeO ratio will be a minimum of 3:1. With a maximum surface roughness of 10 μ inch and fewer than 40 distinguishable pits, inclusions or defects visible at 3500X magnification, this is the smoothest tube in the TrueTube family. O’Brien TrueTube EP provides improved corrosion resistance and reduced dry-down time.

Not All EP Tubing is Equal
Specifying electropolished tubing without specifying corresponding requirements for Cr:Fe, CrO:FeO and SEM defect count as well as surface roughness may not provide a tube surface capable of improving sample transport performance. Following is a side-by-side comparison of TrueTube EP that meets the specification and a tube that was simply specified and supplied as electropolished. While surface roughness is low, the SEM defect count was too high to measure. While the surface roughness in the competitor’s product is low, the SEM defect count was too high to measure.

<table>
<thead>
<tr>
<th>Average Ra (μin)</th>
<th>O’Brien Analytical</th>
<th>Competition</th>
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</thead>
<tbody>
<tr>
<td>Micrograph</td>
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<tr>
<td>1</td>
<td>14</td>
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<td>5</td>
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<tr>
<td>Average</td>
<td>11.8</td>
<td>N/A</td>
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</tbody>
</table>

Footnote from RJ Lee report:
* The surface is not characteristic of high purity electropolished stainless steel, and defect counts would be meaningless.

TrueTube EPS
The ultimate product for corrosion resistance and aversion to sulfur compounds. TrueTube EPS combines the advantages of electropolished and SilcoNert 2000 elemental silicon coated tubing to produce a superior tube. Electropolishing provides the best possible surface for the SilcoNert coating process. The TrueTube EPS combination delivers results that dramatically outperform either electropolishing or SilcoNert coating alone.
Consider the following selection guidelines:

<table>
<thead>
<tr>
<th>Tube</th>
<th>General Uses</th>
<th>Characteristics</th>
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</thead>
<tbody>
<tr>
<td><strong>Commercial 316L Seamless SS</strong></td>
<td>Standard instrument tubing. Free Fe will react with most acids.</td>
<td>Average Surface Roughness: +80 μ inch&lt;br&gt;Limitations: Limited by chemical reactivity and oxygen levels. Subject to stress corrosion cracking.&lt;br&gt;High Ra values of internal surface contribute to adsorption / desorption problems.&lt;br&gt;Advantages: Inexpensive and readily available.</td>
</tr>
<tr>
<td><strong>Fluoropolymer (Teflon®)</strong></td>
<td>Low pressure sample and chemical lines where 316 SS is not acceptable. Used for applications requiring cleanliness.</td>
<td>Average Surface Roughness: NA&lt;br&gt;Limitations: Limited by chemical reactivity and oxygen levels. Temperature and pressure limits vary by variety of fluoropolymer. Very permeable. Used for manufacture of permeable membranes.&lt;br&gt;Advantages: Excellent chemical resistance. Flexible.</td>
</tr>
<tr>
<td><strong>TrueTube CP</strong></td>
<td>Analyzer sample lines where sample cleanliness is important.</td>
<td>An O’Brien TrueTube Product&lt;br&gt;Chemically Polished and Cleaned 316L Seamless SS&lt;br&gt;Average Surface Roughness: less than 40 μ inch&lt;br&gt;Limitations: Limited by chemical reactivity and oxygen levels. Subject to stress corrosion cracking.&lt;br&gt;Advantages: Improved surface finish and cleanliness. Free Fe contaminants eliminated</td>
</tr>
<tr>
<td><strong>TrueTube FS</strong></td>
<td>Moderate acid samples and sulfur.</td>
<td>An O’Brien TrueTube Product&lt;br&gt;A secondary SilcoNert™2000 coating applied to chemically polished 316L SS tubing.&lt;br&gt;Average Surface Roughness: NA&lt;br&gt;Limitations: Very poor resistance to bases. Reacts adversely with fluorides.&lt;br&gt;Advantages: Has improved chemical resistance over substrate alone. Can be used at higher temperatures than polymer coatings. Covalently bonded matrix which reduces surface tension. Has found wide acceptance for transporting sulfur samples.</td>
</tr>
<tr>
<td><strong>TrueTube HCR</strong></td>
<td>Basic and highly acidic sample systems where corrosion is a prime concern.</td>
<td>An O’Brien TrueTube Product&lt;br&gt;A secondary Dursan™ coating applied to chemically polished 316L SS tubing.&lt;br&gt;Average Surface Roughness: NA&lt;br&gt;Limitations: Adsorption / desorption sensitivity at ppb and very low ppm concentrations. Avoid exposure to HF.&lt;br&gt;Advantages: Most corrosion resistant coating. Acid / base exposure pH 0 to 14. Two times more wear resistant than bare 316 stainless steel.</td>
</tr>
<tr>
<td><strong>TrueTube EP</strong></td>
<td>Critical sample systems where adsorption / desorption is a problem.</td>
<td>An O’Brien TrueTube Product&lt;br&gt;Electropolished and chemically treated 316L SS seamless tubing.&lt;br&gt;Maximum Surface Roughness: 10 μ inch&lt;br&gt;Limitations: Limited by chemical reactivity and oxygen levels.&lt;br&gt;Advantages: Cr/Fe ratio better than 1.5:1 and CrO/FeO ratio better than 3:1 improve chemical resistance. Improved adsorption / desorption characteristics compared to commercial tubing.</td>
</tr>
</tbody>
</table>
Consider the following selection guidelines:

**Moisture**
- Thermocouple Cleaned (recommended lower effective application approx. 80 ppm)
- TrueTube® CP Chemically Polished and Passivated (recommended lower effective application approx. 40 ppm)
- TrueTube® EP Electropolished (recommended lower effective application approx. 40 ppb)
- TrueTube® EPs Electropolished with SilcoNert™ 2000 (recommended low ppb to ppm)

**Ammonia**
- PFA Fluoropolymer Heavy Wall (recommended lower effective application approx. 10 ppm)
- TrueTube® FS Chemically Polished with SilcoNert™ 2000 (recommended lower effective application approx. 10 ppm)
- TrueTube® EPs Electropolished with SilcoNert™ 2000 (recommended low ppb to ppm)

**Sulfur**
- Thermocouple Cleaned (recommended lower effective application approx. 80 ppm)
- TrueTube® CP Chemically Polished and Passivated (recommended lower effective application approx. 40 ppm)
- TrueTube® FS Chemically Polished with SilcoNert™ 2000 (recommended lower effective application approx. 10 ppm)
- TrueTube® EPs Electropolished with SilcoNert™ 2000 (recommended low ppb to ppm)

**Mercury**
- PFA Fluoropolymer Heavy Wall (recommended lower effective application ppm)
- TrueTube® EPs Electropolished with SilcoNert™ 2000 (recommended low ppb to ppm)

**Hydrogen Sulfide Recovery Data**

**High pH and Abrasive Samples**
- TrueTube HCR Chemically Polished with Dursan™ (recommended lower effective application 10 ppm)
- TrueTube® EPs Electropolished with SilcoNert™ 2000 (recommended low ppb to ppm if compatible with process)