CONCLUSIONS

The selection of a scraping tool depends on the type of residue to be sampled. This study focused on "hard" residues on metal surfaces, as these are the most challenging residues for sampling. If the residue consists only of metal corrosion, any of these sampling methods, including swabs, are suitable. The entire swab head can be examined in an SEM equipped with an EDS detector, and it is possible to detect and identify sub-micron metal particles without interference from the carbon-rich substrate. But if the residue contains some organic material, or if one wishes to perform solvent extractions on the residue to look for the presence of oils, the residue should be removed from the substrate for analysis by IR or other methods.

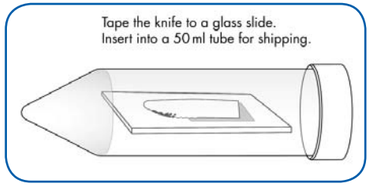
Swabs For small amounts of organic residue, cotton and polyester swabs are poor sampling tools. For residues that consist of purely metal corrosion, swabs can be used.

Razor Blade The razor blade removes a large amount of hard residue deposit, but can scratch the surface of the tank, and is most useful for hard or soft non-metallic residues, as the blade sheds fine metal wear particles that mix with the residue.

Polyethylene/ Polypropylene Tools Soft plastic scrapers such as the polyolefin pipette tips and poly-propylene spatula are useful only for soft waxy residues, since harder residues become entrained in abraded polyolefin debris.

Tape Lift The tape lift method can be useful for hard residues that are not firmly adhered to the metal surface. Solvent rinsing may be needed to remove any adhesive that clings to the particles after they are removed from the tape. The tape cannot be used for soft residues.

Polystyrene Tools The polystyrene scraping tools, including the knives and the plastic cup scraper, were the best choices for hard residues because of their hardness and solubility characteristics. The polystyrene tools are good sampling tools for soft residues, as well. These tools are stiff enough to remove the residue without generating a large amount of abraded plastic, but do not damage the metal surfaces. The knives are especially convenient because very little sample preparation is needed, and they can be obtained in many different colors for a variety of colored residues. It is very easy to remove the residue from the knives with a tungsten needle, and transfer it to another substrate for analysis. Polystyrene is readily soluble in many common solvents; if contamination of the residue with knife debris is suspected, it can be easily dissolved. The infrared spectrum of polystyrene is unique, and it is substantially different from most process tank residues, thus it is easy to check for the presence of polystyrene in the sample, and in most cases the dissolution process should not be necessary.



The knife, or any of the other sampling tools, can be attached to a glass slide with tape as shown, and inserted into a standard 50mL plastic tube with a threaded cap for shipping or safe transport to the lab for analysis.

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Post-Conference Presentation

Comparison of Sampling Methods for Identification of Process Tank Residues

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OBJECTIVE

After routine cleaning of process tanks, residues are sometimes observed on the tank surfaces, appearing as faint whitish or dark stains that cannot be easily removed. It is desirable to identify the residue so that it can be removed without damaging the tank surface. Due to the small amount of residue on the surface, it can be difficult to obtain enough sample for analysis. For unambiguous identification of the residue, it is necessary to obtain a pure sample of the residue without any attached material from the sampling tool. The most common sampling tool has been a cotton or polyester swab, sometimes soaked in water or alcohol. Swabs are poor sampling tools because only a small amount of residue is transferred to the swab, the sample is difficult to remove from the swab for analysis, and light-colored residues may not be seen on the swab. Our objective was to develop an alternative sampling method to obtain a larger sample of residue that could be easily isolated for micro-analysis by FTIR or EDS. The sampling methods are not limited to tank surfaces, as they can be applied to any stained solid surface.

BASIC PROCEDURE

The stained surfaces consisted of a flat stainless steel sheet having a thin spotty coating of rust, and a stainless steel vessel having a faint coating of hard water deposits. Stained surfaces were vigorously rubbed with the dry swabs, or moderately to lightly scraped with the other sampling devices while holding the device at about 45 degrees to the surface. The tape lift was obtained with a single firm compression of the tape against the stained surface. The sampling devices were examined and photographed at magnifications up to 25X. In some cases, a tungsten needle was used to remove some of the residue from the sampling device, then the residue was transferred to a glass slide and examined using a polarizing light microscope at 500X magnification.

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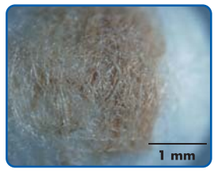
Comparison of Sampling Methods for Identification of Process Tank Residues

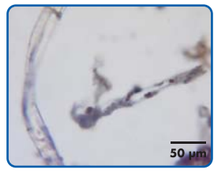
SUPPLIES

- Cotton swabs
- Polyester swabs
- Razor blades (Personna®, double-edged)
- Scotch® tape, double-sided
- Polyethylene disposable pipette
- Polypropylene pipette tips(Thermo-Scientific™ Finntip™ Flex™ 300)
- Polypropylene plastic spatulas (VWR® Scientific Catalog #80081-188)
- Scraper made from a clear polystyrene plastic cup
- Polystyrene plastic knives (various colors and sources)
- Stereomicroscope equipped with transmitted, coaxial, and oblique light sources
- Polarizing light microscope
- Glass slides
- Slide storage box
- 50mL plastic vial

COTTON SWAB

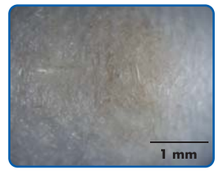


When this swab is used to sample a rust-colored surface, the residue is readily visible, but is trapped between and below the swab fibers, making it very difficult to isolate the brown material without removing some cotton as well.

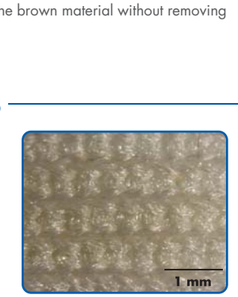


Several fibers were removed from the brown area of the swab and examined using a polarizing light microscope. The brown residue cannot be separated from the fibers for analysis.

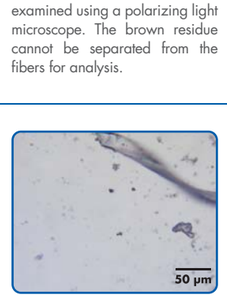
When a cotton swab is used to sample a light-colored residue, no residue is visible on the swab with the naked eye. At 25X, a faint residue is seen on the swab, but it is impossible to isolate the residue from the fibers. A white residue may not be seen, even at higher magnification.



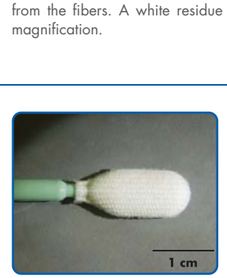
POLYESTER SWAB



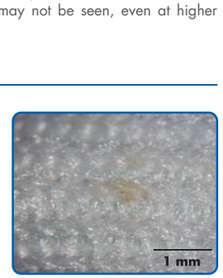
The dry swab was used to sample a rusty steel surface. The rusty residue is not visible as discrete particles, and cannot be removed from the swab for analysis.



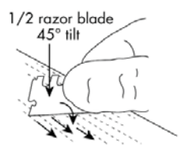
Several fibers were removed from the brown area of the swab and examined using a polarizing light microscope. A few loose rust particles were dislodged from the surface of the fibers, but this is not a reliable sampling method.



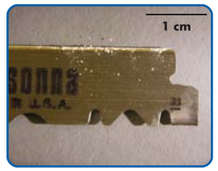
Light-colored residue is almost invisible on the polyester swab, even at higher magnification. A white-colored residue would not be visible on this swab, even at higher magnification.



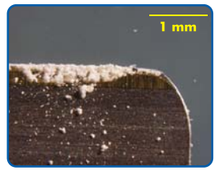
RAZOR BLADE

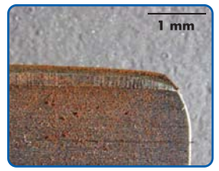


Double-edged blades were cut in half with metal shears, and held at a ~45 degree angle while gently scraping the surface.



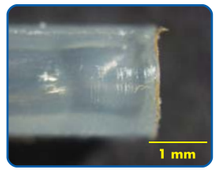
Light-colored residue is clearly visible without a microscope.

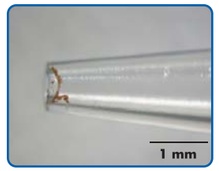




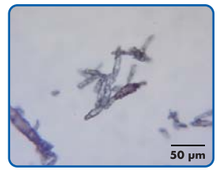
Light-colored and dark-colored residues are easily seen on the blade, and can be easily removed for analysis. The blade may scratch some sample surfaces, and fine metal wear particles can be dislodged from the blade and mixed with the sample, making it difficult to identify residues that consist of metal corrosion.

POLYETHYLENE AND POLYPROPYLENE PIPETTE TIPS



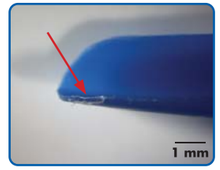


Dark-colored residues are visible on colorless polyolefin scraping tools. Light-colored residues would not be readily visible. Further, polyolefin plastics are very soft, and tend to form rolls of plastic with the particles of interest entrained within.

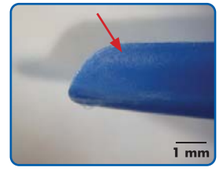


Some of the collected material was removed from the polypropylene pipette tip and examined using a polarizing light microscope. The brown residue cannot be separated from the loose plastic filaments for analysis.

POLYPROPYLENE SPATULA

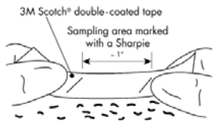


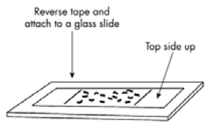
Light-colored residues are easily seen on the dark-colored spatula. However, the soft polypropyleneforms rolls of plastic with entrained particles of residue.

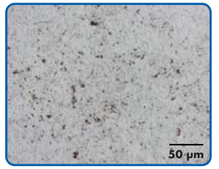


Some loose particles of residue collected on the surface of the trough, not immediately in the area of contact of the spatula with the metal surface. The loose residue clings to the spatula, and can be easily removed for analysis. However, in general, this type of scraping tool is best suited for soft waxy residues, not hard deposits.

SCOTCH® TAPE

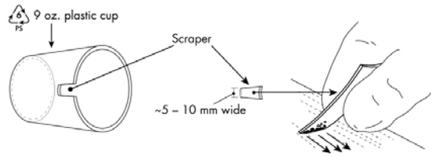


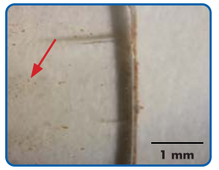




The tape picks up a good sampling of residue, but the particles must be removed from the adhesive and cleaned with a solvent to remove any attached adhesive. This can be difficult if the particles are very small or crumbly. The tape lift is only useful if the residue is not firmly adhered to the surface.

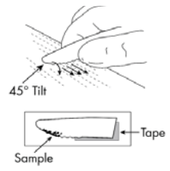
POLYSTYRENE SCRAPER (CUP)



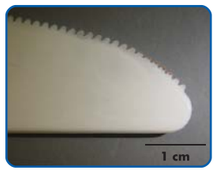


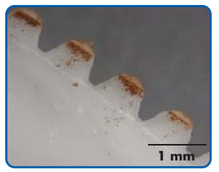
The scraper collects a scattered deposit of loose particles which can be removed for analysis. Most of the residue on the cut edge of the scraper is mixed with loose polystyrene flakes. Some of this residue can be transferred to a glass slide and the polystyrene can be dissolved with a solvent if necessary, freeing the residue for analysis.

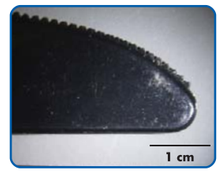
POLYSTYRENE KNIFE

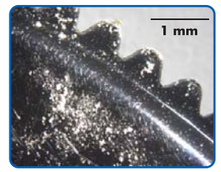


The handle is removed from the knife so that the scraping edge can be held as shown, and heavy pressure can be applied as the knife edge is scraped over the surface. The end of the knife that does not contain the sample can be attached with tape to a glass slide for easy transport to the lab.









The teeth of the knife collect a good sampling of residue, and a large amount of loose residue clings to the surface of the blade adjacent to the teeth. The loose residue can be easily removed for analysis. The residue on the teeth contains some fine polystyrene particles that were abraded during the scraping process, which can be dissolved with a solvent if necessary. Polystyrene knives are inexpensive and can be obtained in a number of colors for use with different colored residues.