OIL TESTING KIT

Introduction

The **Oil Testing Kit** is an open source Do-It-Yourself kit which attempts to make it possible to identify oil pollution by type. This means matching a suspected sample with a known sample of crude oil, motor oil, heating oil, or other petroleum-based contaminant using a homemade fluorescence spectrometer. A spectrometer enables you to precisely measure the colors of light emitted by carefully prepared samples when they are illuminated with strong ultraviolet light, as shown above.

Kickstarter

We're running <u>a Kickstarter campaign</u> to distribute a run of these kits. Watch this video to learn a bit about it, and about the Public Lab community:

Collect, Scan, Compare

The process of testing for oils can be described in three overall steps;

- 1. collecting samples of suspected oil or tar from the ground, and dissolving small amounts in mineral oil so they are transparent
- 2. illuminating the solutions with ultraviolet light -- presently using a 405 nanometer blue laser -- and recording the light spectrum with a DIY spectrometer
- comparing the spectrum to those of similarly prepared samples of <u>known pollutant oils</u>, as well as a negative control

Here we will discuss and illustrate these steps in depth -- but keep in mind this process is always evolving. See the <u>Challenges section</u> for ways to get involved and contribute.

Collect

Locating samples

Originally, we focused on tar balls which were washing up on US Gulf Coast shorelines following the BP oil spill. These ranged from hard black lumps to orange residue. But oil contamination takes many forms, from residue around a street drain, to a sheen or buildup on the surface of the water. Here are some examples:



Left to right: dried oil on rocks in 2010, Louisiana coast <u>by Cesar Harada CC-BY-NC-SA</u>, oil residue in the ocean in 2010, Louisiana coast <u>by Cesar Harada CC-BY-NC-SA</u>, Oil tanker leak <u>on tracks beside Mississippi</u> <u>River</u>, by <u>@marlokeno</u>, swabbing a street grate by <u>@warren</u>

Labeling

Label sample bottles with the date, time, and location. If you also give it a unique number, any other information can be kept in a notebook next to that number, such as further notes on the location and its condition. Take a photo of the sample with your label, in the place you found the sample, for context.

This photo is of a sample already dissolving in mineral oil, but typically we've collected relatively dry samples and dissolved them later. You can put a small amount into an empty sample jar or use the cue tips to put residue directly into mineral oil as in the next step.



Preparing samples

Use a cotton swab or small brush, dipped in mineral oil, to break up some of the material and dissolve it in a small, square-sided glass jar of mineral or baby oil. Wear gloves before handling suspected pollutants. You may need to rub the sample for a while to get it to dissolve. If it <u>does not dissolve</u>, <u>there may be more</u> <u>aggressive ways to dissolve it</u>. Where possible, try not to put too much sand or other stuff in the jar. It's a good idea to keep extra samples (dry, as you found them, not in mineral oil) in glass jars, stored in a cool dark place, as there may be an opportunity to test them later with more expensive, official means (see <u>Validate your</u> <u>results</u> below).



Seal the bottle tightly with the cap. You can then gently turn it over a few times to get the residue to dissolve -it may take some time before the mineral oil takes on a distinct but faint yellowish hue. You may then have to wait for the sediment to settle out. You want the liquid to be quite transparent, with the chunky stuff settled to the bottom.

Concentration

One big issue is getting the correct concentration of sample dissolved. If it's too little, we may not be able to get it to glow under UV light. Too much and it could be too dark for the light to be visible in the bottle. Ideally we'd like to have the same concentration in each sample bottle, but determining this is very difficult as the samples may be mixed, so they can't easily be weighed. We recommend going by how dark they are -- try for a color similar to very dilute tea:



This sample by <u>@eustatic</u> is too dark for the laser to get through:

http://farm4.staticflickr.com/3725/9711572372_3aa638cf46.jpg

Scan

Now that your sample is prepared, you may be able to get it to fluoresce or glow by shining an ultraviolet light through it. We have had good results using a blue/UV laser, a 405 nanometer laser which is the same as found in a Blu-Ray player. See <u>the parts list</u> below for where to buy one. Strong UV LEDs can also work, but are not as bright. They are, however, easier to line up with a spectrometer's opening slit. LEDs are also not as narrow wavelength as a laser.



Don't look at the laser too much, as it can hurt your eyes, even if you're not pointing it directly at your eye!

Note that the laser will have a purple-ish color by itself (as seen in the lead image at the top of the page) -- this is not fluorescence, but just scattering of the laser light. What you're looking for is any other color -- whitish, bluish, greenish -- which is not from the laser, but is produced in the material itself as it's excited by the UV light.

To measure precisely the colors that are being produced, we will use a spectrometer.



Spectrometry

Colored light is often a blend of different colors. A spectrometer is a device which splits those colors apart, like a prism, and measures the strength of each color. A typical output of a spectrometer looks like this **spectrum of the daytime sky**, with the actual light spectrum at the top and the graph of wavelength (horizontal axis) and intensity (vertical axis) below:



While there are many ways to use a spectrometer, in this case we're causing the samples to glow by exciting them with a high-energy UV light. When we scan the fluorescence from an oil sample, we can clearly see the laser color, or wavelength, which is only in a narrow range around 405 nanometers, to the left:



All the remaining light, to the right of that tall peak, is produced by the excited material in the sample. The **shape of that curve** can be matched against other samples to help us identify what ours is. But first, we'll need a spectrometer.

This section is under construction.

Construct a spectrometer

Public Lab first began developing cheap spectrometers in the winter of 2010, after the Deepwater Horizon oil disaster in the Gulf of Mexico. Since then <u>we've made enormous progress</u> and have a variety of designs for less than \$100, including:

- a USB webcam based kit (plans)
- a molded plastic smartphone-based kit (plans)

• a very affordable foldable papercraft kit which attaches to a smartphone or webcam (plans)

Of course, the plans for all of these are on the Public Lab website and are open source hardware designs, so you can always make your own -- the kits are intended to help you get started.

However, **none of these designs currently includes a place to put a sample, or a laser to illuminate it** -they were designed before we developed that technique. So we are currently working on a new version, and need your help! See the<u>Challenges</u> section for more ways to get involved.

For now, you can purchase <u>the add-on "Alpha" Oil Testing Kit</u> (see <u>parts list</u> to assemble your own) in combination with the existing <u>Desktop Spectrometry Kit</u>, which is what was used in the examples on this page. See <u>the main spectrometer page</u> for build instructions.





A variety of prototypes for scanning fluorescence in oil samples.

Illuminate the sample

Whether you use the Alpha Oil Test Kit, or one of the <u>prototype designs</u> we've <u>recently published</u>, the basics are that you need to illuminate your sample with a laser beam perpendicularly to the direction your spectrometer is pointing, and to align it so that you can see enough light using the software at <u>SpectralWorkbench.org</u>, which connects to your USB webcam. The illustrations above show a couple ways we've folded up or modified boxes to keep a spectrometer lined up with a sample container and a jar.

The hard parts are getting the laser lined up with the slit, so the light actually goes into the spectrometer, and using a pretty sensitive webcam so that it can actually detect the light. One thing not shown as clearly above is that you should either turn the lights off or cover the box so that you don't detect ambient light from the room.



The illustration shows scanning a control sample at the same time, but this may not be necessary if everything is consistent between scans. You'll want to see something like this in the software:



If you don't, but you can visibly see fluorescence (<u>see below</u> for examples), try moving the laser up and down a bit to get it to align. You want the curve to the left of the tall peak (which is the laser) to be mostly between 25% and 75% intensity, so it's not "clipping" by being too bright but you're getting enough light to see a clear shape amongst the noise. You should also use "RGB mode" (in the Tools section of a saved spectrum page) to check that none of the three channels is overexposed. We'll add an automated warning for overexposure, soon:



This process of getting enough light into the spectrometer needs improvement -- see the <u>Challenges</u> section to help out.

Refine your technique

Once you get a basic scan, save and label it, but consider some of these techniques to improve your data collection:

- try to ensure the same concentration of contaminant in each sample bottle (see <u>Challenges</u> for more on this)
- take several scans for each sample, and label them #1, #2 -- comparing them to ensure your technique is consistent
- scan multiple samples from the same site
- smooth your data using the macro described in this note

Once you're confident that your sampling is consistent and rigorous, you're ready to start comparing the data you've collected.

Compare

When identifying an oil, we are hoping to measure the color of fluorescence of the Poly-Aromatic Hydrocarbons (PAHs) in the sample. The best way to identify a sample would be to compare it to a selection of similarlyprepared known reference materials. For example, if you have unknown X, you could compare it to both: A) a **known sample of crude oil** and B) a**known uncontaminated sample of material** (perhaps soil) to see which it matches best.



Which is it more like? Ideally, it should be compared to a range of possible references. For example, if it's possible the sample is heating oil or motor oil, you could compare it to similarly prepared samples of those as well. <u>Some research has shown</u> that vitamins A and E can produce fluorescence similar to petroleum products.

Read over <u>this detailed research note</u> to see how to set up a comparison -- but keep in mind that since it was published, we've vastly improved noise reduction (smoothing) and comparison features <u>as described in this note</u>.





Plot your samples and compare

Add all your scans to a set, so they can be viewed together, and you can see the subtle color differences as graphed lines. Add the spectrum of your unknown sample and see which of the others it is closest to.

Calibration and intensity

Your spectrometer <u>should be calibrated</u> and the very tall peaks from the laser light should align if this has been done correctly. If your scans are too dim (mostly under <25%) or too bright (hitting the 100% ceiling), you may want to try re-running them.

If your scans were not made with the same amount of light, or if the spectrometer was not aligned the same way, two scans of the same material may not appear the same on your plot -- which would make it hard or impossible to see your sample is a good match or not. <u>Some research suggests</u> that such spectra should be comparable if their area is equalized -- this <u>remains to be demonstrated</u>

Positive and negative controls

Think critically about your testing and how it might have gone wrong. Could you have made mistakes, or is the match you've found between your unknown sample and your references not good enough? Could another material produce the same color spectrum as your suspected contaminant, and fool your test? (See <u>this</u> research on Vitamins E and A causing such false positives).

Validate your results

An extra step that may give your work more credibility is to submit a few of your samples for analysis to a lab, or to <u>use other tests</u> to confirm your results. Alternatively, if you know other testing has occurred, you can try to extend its results by re-testing the same site or samples, correlating your results with the previous test, and performing your own tests over a larger area or on more sites, or over a longer time span.

Publish

Ask others to critique your work or help you refine it <u>on the plots-spectroscopy discussion list</u> or by <u>posting a</u> <u>research note thoroughly describing your results</u>. Even if your work is not done, it's a great idea to share and solicit feedback on your plan before, during, and after you've done the work. You may be able to build on previous work on the website, and your work will help others who are seeking to perform similar tests.

Dissolving samples

We use mineral oil as it's non-toxic and cheap, and can be purchased in most pharmacies as either mineral oil or "baby oil". However, some samples may be hard enough that they don't dissolve readily, and more aggressive solvents may be able to dissolve these, such as methanol or denatured alcohol. These are not as safe to handle, however, so we advise caution if you attempt this. **Please** <u>post a research note</u> if you attempt this, as it is an unexplored area.

Challenges

This document, and this methodology, is still under active development. What you see on this page is only the best attempt so far at collating and presenting the <u>work of Public Lab contributors to date</u>. Some of the challenges that remain include:

- developing a better, simpler method for getting consistent concentrations of sample dissolved
- determining if <u>normalizing the area under the curve</u> (in software) for different samples may make consistent concentration less important -- can matching still succeed?
- developing a quick-to-assemble but precise and durable and cheap version of <u>Public Lab's DIY</u> spectrometer for scanning samples
- scanning known samples of oil for use in comparisons
- determining if it's necessary to scan references (and have crude samples) when testing, or if we can rely on previously scanned references -- are scans consistent enough or do we have to <u>mail crude around</u>?
- exposure: how do we ensure enough light from the sample enters the spectrometer, and (when using a laser) that aligning the light with the slit is easy?
- developing a way to save smoothed data in SpectralWorkbench

Be sure to <u>share your research</u> as you tackle these questions -- publish early and often! Remember that every additional step can add complexity and cost to the process, so always keep in mind what such steps achieve, and balance that against the potential barrier to entry they cause.

Variations

There are many variations of the process which could be useful but are not essential. These include:

- collecting samples from a sheen on the surface of the water -- which may be difficult as sheens are extremely thin and spread out
- measuring fluorescence in-situ on the ground, without collecting or concentrating samples in a jar -- which could be difficult as it's very dilute and mixed with other things like water, dirt, or plant matter

Many of these may be future goals of the project, but we are focusing on our primary use case of collecting contaminated soil or residue from the ground, dissolving it in mineral oil, and illuminating it with UV in a spectrometer.

Source : http://publiclaboratory.org/wiki/oil-testing-kit