BLACKSTONE LABORATORIES

The Oil Report
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Oil the News that's Fit to Print!

Did you hear? Our new labels speed up delivery time for your sample. If your black mailers have the old, Merchandise Return label on them, click here and we'll send you our new Tyvek envelopes with the new label attached!

Sample Methods
Are you doing it wrong?

by Amanda Callahan

It's a perfect spring day. There you are, merrily going about your business of changing the oil. But wait! You forgot the oil sample bottle! A quick scramble to retrieve the bottle gets you back to the oil just as the last of it drains out.

Can you pour a sample out of the filter instead? What if you add a quart a few days before sampling – how does that affect the analysis? What about something like an engine flush – should you use one? Do they work? Your investigative team at Blackstone experimented, and we've got answers. While these tests probably won't qualify for a peer-reviewed journal, they're a good guide to what you need to know about sampling.

This is part two in our series on sampling methods. Part one, on engine flushes and their effects on analysis, can be found here. This article covers common sampling scenarios: does it change the results if you take a sample from the filter or dipstick tube? What if you add fresh oil before sampling? Is it a problem if the oil gets dark right away? That last question isn't about sampling methods, but people ask all the time and your investigative team at Blackstone wanted to know, so read on for answers.

Does it matter how you sample?
Our instructions for sampling say to catch a sample as the oil drains from the pan, but that doesn't always happen. Does it change the data if you take a sample from the filter or pull it through the dipstick tube?

Sampling a Toyota Corolla		Dipstick	Pan	Filter
	MI/HR on Oil	8,053	8,053	8,053
	MI/HR on Unit	32,141	32,141	32,141
	Sample Date	10/10/2021	10/10/2021	10/10/2021
	Make Up Oil Added			
ELEMENTS IN PARTS PER MILLION	ALUMINUM	3	4	4
	CHROMIUM	1	1	1
	IRON	12	12	13
	COPPER	**23**	**22**	**23**
	LEAD	0	0	0
	TIN	0	0	0
	MOLYBDENUM	531	514	538
	NICKEL	0	0	0
	MANGANESE	2	2	2
	SILVER	0	0	0
	TITANIUM	4	4	4
	POTASSIUM	2	2	2
	BORON	77	75	78
	SILICON	**41**	**40**	**47**
	SODIUM	5	5	5
	CALCIUM	1299	1269	1328
	MAGNESIUM	588	570	596
	PHOSPHORUS	748	737	773
	ZINC	879	869	913
	BARIUM	0	0	0

Fig 1 - Sampling a Corolla from the dipstick tube, pan, and filter

In short: no. Figures 1 and 2 illustrate three consecutive samples taken from two different cars: Figure 1 is from a Toyota Corolla and Figure 2, a Mercury Milan.

The column on the left is a sample taken through the dipstick. The middle column was oil taken while the oil drained from the pan. And the right-hand column is oil taken from the filter.

Results
The samples are unremarkable in that there's less than 1 ppm difference in the wear metals across all three samples. The sampling method seems to have no impact on the metals that show up in analysis.

The Corolla in Figure 1 does show a higher silicon reading in the sample taken from the oil filter, but perhaps that was due to either dirt collected by the filter that ended up back in suspension in the engine oil, or sample contamination – we did have to use a bit of creativity in removing that filter from the engine, as the filter was overtightened and stuck. (If you're wondering, we stabbed it with a screwdriver to give us more

Sampling a Mercury Milan		Dipstick	Pan	Filter
	MI/HR on Oil			
	MI/HR on Unit			
	Sample Date	10/18/2021	10/18/2021	10/18/2021
	Make Up Oil Added			
ELEMENTS IN PARTS PER MILLION	ALUMINUM	4	4	4
	CHROMIUM	1	1	1
	IRON	14	14	14
	COPPER	0	0	0
	LEAD	0	0	0
	TIN	0	0	0
	MOLYBDENUM	142	144	142
	NICKEL	0	0	0
	MANGANESE	0	0	0
	SILVER	0	0	0
	TITANIUM	0	0	0
	POTASSIUM	0	0	0
	BORON	18	18	18
	SILICON	12	12	12
	SODIUM	4	4	4
	CALCIUM	1482	1498	1488
	MAGNESIUM	473	477	473
	PHOSPHORUS	634	642	638
	ZINC	750	758	753
	BARIUM	0	0	0

Fig 2: Sampling a Mercury Milan from the dipstick tube, pan, and filter

twisting leverage – we did not sterilize the screwdriver before surgery, so it's entirely possible some silicon was introduced in that process.)

Does adding fresh oil impact the test results?
It makes sense that adding fresh oil will dilute the wear numbers. But how much do the numbers change? And does it matter *when* you add the new oil? In theory, if you have a 4-quart sump, adding one quart of fresh oil shortly before the oil change would mean that your engine's metals are diluted by 25% from their previous numbers.

To test this theory, Ryan Stark, Blackstone's president, pulled a sample from his MINI, then added a quart and sampled again to see how the numbers changed (see Figure 3).

Crunching numbers
The MINI has a total capacity of 4.5 quarts, so the one quart he added comprised 22% of the total engine oil capacity. Most of the metals decreased by approximately the same percentage: iron dropped from 26 ppm to 20 ppm (a decrease of 23%), copper dropped by 25%, from 8 to 6 ppm. If we assume that chrome actually changed by less than 1 full ppm, due to rounding, the average change in metal works out to around 25%, which is what we'd expect from adding a quart of oil to this engine.

The only other appreciable wear metal in his sample is aluminum, which, interestingly enough, read at 5 ppm in both samples, showing no change at all. We couldn't let that element go without a little suspicion – why didn't it change when the other metals did? As it turns out, the actual number our spectrometer reads goes four decimal places to the right. We round to the nearest whole number on the report, but if we pull the full spectral data from those tests, aluminum read at 5.4290, and in the second test aluminum read at 4.8995. Both readings were rounded to 5 ppm in the report, but the full spectral data shows a slight change between the two samples, an improvement of 9.7%. So aluminum did change with the added oil, just not quite as much as the other metals and not enough to show on one of our published reports.

The "when" factor
There are other variables to consider like how far into your oil change you add the oil, and how much oil you add. If a quart of oil is added at the 3,000-mile mark and you run your oil 10,000 total miles, the dilution factor probably is going to be a lot different than adding a quart just before changing the oil. That's harder to test for because there are too many variables to isolate.

MINI Cooper		Before adding a quart	After adding a quart
	MI/HR on Oil	3,972	3,972
	MI/HR on Unit	53,734	53,734
	Sample Date	11/7/2021	11/7/2021
	Make Up Oil Added	1 qt	2 qts
ELEMENTS IN PARTS PER MILLION	ALUMINUM	5	5
	CHROMIUM	1	0
	IRON	26	20
	COPPER	8	6
	LEAD	0	0
	TIN	0	0
	MOLYBDENUM	81	98
	NICKEL	1	1
	MANGANESE	1	1
	SILVER	0	0
	TITANIUM	0	0
	POTASSIUM	0	0
	BORON	80	82
	SILICON	6	6
	SODIUM	4	4
	CALCIUM	1075	1122
	MAGNESIUM	626	605
	PHOSPHORUS	772	743
	ZINC	858	857
	BARIUM	0	0

Fig. 3: Before and after adding oil to a MINI Cooper

So this isn't the be-all-end-all of the dilution question, but it at least gives some insight into the fact that the metals could be diluted if you're adding oil, especially if you're doing it right before an oil change. It is a good idea to add fresh oil when low, even if you'll be changing the oil soon. Running an engine on a diminished oil capacity isn't great.

Why does my used oil look so dark?
We get a lot of questions from people who do an oil change then notice that their oil is dark immediately afterward. Is it a problem?

To get to the bottom of this question, we conducted two oil changes on two separate vehicles, idled the fresh oil for five minutes, then sampled and examined the new oil.

In both cases, the oils were quite dark after just five minutes of use. In Figures 4 and 5, the virgin oil is pretty obvious, but there's not much difference between the new oil with 5 minutes on it and the oil with several thousand miles on it. In terms of the overall sample color, it's quite hard to tell.

Results
So does the dark oil indicate anything? Figures 6 and 7 show the analytical results of the new (but darkened) oil after being run 5 minutes in two different engines.

Both oils look very clean in testing, with minimal insolubles, no contamination, and very low metal counts. You might note that the metals do not start at 0 ppm – that's because you never get 100% of the old oil out when you do an oil change.

Figures 6 & 7: Analysis on oil run 5 minutes

Corolla: New oil after 5 minutes

	MI/HR on Oil	0
	MI/HR on Unit	32,141
	Sample Date	10/10/2021
	Make Up Oil Added	
ELEMENTS IN PARTS PER MILLION	ALUMINUM	1
	CHROMIUM	0
	IRON	2
	COPPER	3
	LEAD	0
	TIN	0
	MOLYBDENUM	124
	NICKEL	0
	MANGANESE	0
	SILVER	0
	TITANIUM	0
	POTASSIUM	0
	BORON	74
	SILICON	11
	SODIUM	2
	CALCIUM	944
	MAGNESIUM	606
	PHOSPHORUS	567
	ZINC	669
	BARIUM	0

PROPERTIES	SUS Viscosity @ 210°F	48.9
	cSt Viscosity @ 100°C	6.92
	Flashpoint in °F	395
	Fuel %	<0.5
	Antifreeze %	0.0
	Water %	0.0
	Insolubles %	0.0
	TBN	6.1
	TAN	
	ISO Code	

Figure 6

Milan: New oil after 5 minutes

	MI/HR on Oil	0
	MI/HR on Unit	
	Sample Date	10/18/2021
	Make Up Oil Added	
ELEMENTS IN PARTS PER MILLION	ALUMINUM	1
	CHROMIUM	0
	IRON	2
	COPPER	0
	LEAD	0
	TIN	0
	MOLYBDENUM	46
	NICKEL	0
	MANGANESE	0
	SILVER	0
	TITANIUM	0
	POTASSIUM	0
	BORON	119
	SILICON	7
	SODIUM	1
	CALCIUM	778
	MAGNESIUM	668
	PHOSPHORUS	576
	ZINC	660
	BARIUM	0

PROPERTIES	SUS Viscosity @ 210°F	55.5
	cSt Viscosity @ 100°C	8.93
	Flashpoint in °F	430
	Fuel %	<0.5
	Antifreeze %	0.0
	Water %	0.0
	Insolubles %	TR
	TBN	4.6
	TAN	
	ISO Code	

Figure 7

Fig. 4: Toyota Corolla samples

***Left*: Virgin oil. *Center*: oil run 5 minutes. *Right*: oil run several thousand miles.**

Fig. 5: Mercury Milan samples

***Left*: Virgin oil. *Center*: oil run 5 minutes. *Right*: oil run several thousand miles.**

There's always some carryover from one oil change to the next, and you can see that in the results.

So is it a problem that the oil looks dark right after an oil change? Nope. It's fairly normal for oil to darken quickly after an oil change. If anything, it seems to suggest that the oil is doing just what it's supposed to be doing: collecting contaminants and combustion by-products and keeping them in suspension so they can be removed when the oil is changed.

Sampling Methods: Go for it!
In the end, although we give you guidelines about how to sample, your method really doesn't make too much difference. If you don't catch a sample mid-stream, just let us know when you send the oil in and we'll take that into account when we do the analysis. If anything unusual shows up and we think it might be related to something you did, we'll let you know in the comments.

Report of the Month

This 2000 Expedition has a problem. What is it?

To learn where the elements are coming from, click here and scroll down.

UNIT

MAKE/MODEL: Ford 4.6L V-8
FUEL TYPE: Gasoline (Unleaded)
ADDITIONAL INFO:

OIL TYPE & GRADE: Motorcraft Semi-Synthetic 5W/20
OIL USE INTERVAL: 6,828 Miles

COMMENTS

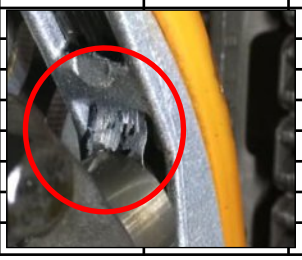
JIM: There's a lot of aluminum in this sample (4.6L V-8 averages are based on a ~5,300-mile run). Excess aluminum in these engines can be from a timing chain tensioner problem, and that may be the case for yours. We're not sure if that would be of any relation to the puff of smoke you see every now and then, but this level of aluminum is cautionary so we suggest you have the engine inspected as soon as possible before a potential failure occurs. Note copper (brass/bronze) and silicon (usually from either sealer/lube or dirt) are also elevated. Resample in 3K miles if all is well.

ELEMENTS IN PARTS PER MILLION

		UNIT / LOCATION AVERAGES		UNIVERSAL AVERAGES
MI/HR on Oil	6,828		4,784	
MI/HR on Unit	232,669		92,012	
Sample Date	4/5/2021		2/10/2005	
Make Up Oil Added	3 qts		0.5 qt	
ALUMINUM	**386**	4	4	4
CHROMIUM	1	1	1	1
IRON	26	15	15	15
COPPER	**17**	12	12	4
LEAD	1	0	0	2
TIN	0	0	0	1
MOLYBDENUM	23	5	4	67
NICKEL	1	1	1	1
MANGANESE	1	0	0	2
SILVER	0	0	0	0
TITANIUM	2	0	0	2
POTASSIUM	0	0	0	2
BORON	82	0	0	58
SILICON	**53**	7	7	15
SODIUM	8	4	4	40
CALCIUM	1543	1761	1761	1917
MAGNESIUM	787	49	49	237
PHOSPHORUS	820	772	772	710
ZINC	956	974	974	828
BARIUM	0	0	0	1

The left side of the timing chain is not riding on the guide, it's down *in* the guide.

The timing chain tensioner is clearly worn on one side.



Left: Metal shavings on the underside of the guide. No bueno.

The owner writes: Over the winter my low oil pressure light stayed on after a cold start for about 10 seconds - this was really the reason I decided to send in an oil sample. Then again in April on a return trip home, my low oil pressure light came on twice for about 3-4 seconds each time. At this point I had already received your report and knew I had to do something. With 233K on the clock, my motor was stone quiet, ran great, and from what I had been reading, I really didn't think my tensioners/chains were the problem.

Anyway, reluctantly I started the process of tearing it down, knowing I'd be fighting 21 years of living in the rust belt, in search of the problem. Took me about 10 hrs to get the oil pan off thanks to all the pan bolts being rusted and rounded off. Finally got the pan off, very little "slime" in the bottom of the pan but when you touched it, you could tell it was aluminum 'paste'. Oil pickup was clean - I was starting to worry as I was expecting to find a plugged oil pickup. Took me about 12-13 hrs to get both valve covers off as almost half the studs and bolts were a rusted mess (I had to weld nuts to them to get them off).

Once I had the covers off, everything looked intact, but I did notice the tensioner arms looked like maybe the plastic was worn. Had the front cover off in about 30 min and as you can see from the pics, my tensioners still had tension on the chains, but the chains had worn through the arms, and actually were starting to wear into the tensioner pistons. The chains looked pretty good (we estimated they had stretched maybe 1/4" total). The guides looked brand new too, and I thought for 233k miles the engine was extremely clean inside. The DS tensioner piston had a slight amount of side to side play in it, and if you squeezed it moderately hard, you could depress it about 1/4".

I'm hoping this was the cause of my intermittent low oil pressure light (either that or my pressure sensor switch is on its way out). I replaced the chains, guides, arms, tensioners and oil pump (for peace of mind). Got it all buttoned up and after priming the oil pump, it started right up. All back together now, and runs great with no leaks! Call me crazy, but I think the engine is even quieter now! Thanks for the great heads up in my oil report, without it I would have kept driving it, as there was no indication anything was wrong.