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Crankcase lubricant formulation

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Outline

- Introduction
- Example of a formulation problem
- Formulation balance
- Formulation aspects
- Engine tests
- Product development program
- Codes of practice
- Summary

Introduction

- In a typical crankcase oil formulation development, the oil must meet performance targets across a wide range of engine and bench tests
  - It is not unusual for a European product development to meet targets in 50 tests (e.g. 25 engine tests + 25 laboratory tests)
- Each test requirement typically demands something different from lubricant performance
  - Thus the formulations ideally suited to one test are not necessarily the same as for another
- The challenge for the formulator is to develop formulations that can meet all of the test/target requirements together
  - We illustrate this in the following formulation example

Example of a formulation problem (1)

Suppose that, in a formulation development*

- We need to pass three tests: **Test 1, Test 2, Test 3**
- For this particular product type, we traditionally use only two additive components: Alpha and Beta
- In scoping work we can identify combinations of Alpha and Beta that can pass **Test 1** and **Test 2** together
  - However none of these combinations can pass **Test 3** and this is a major issue for the project
- This is illustrated in a plot in the next slide

*(This example is highly idealised in order better to illustrate principles of formulation)

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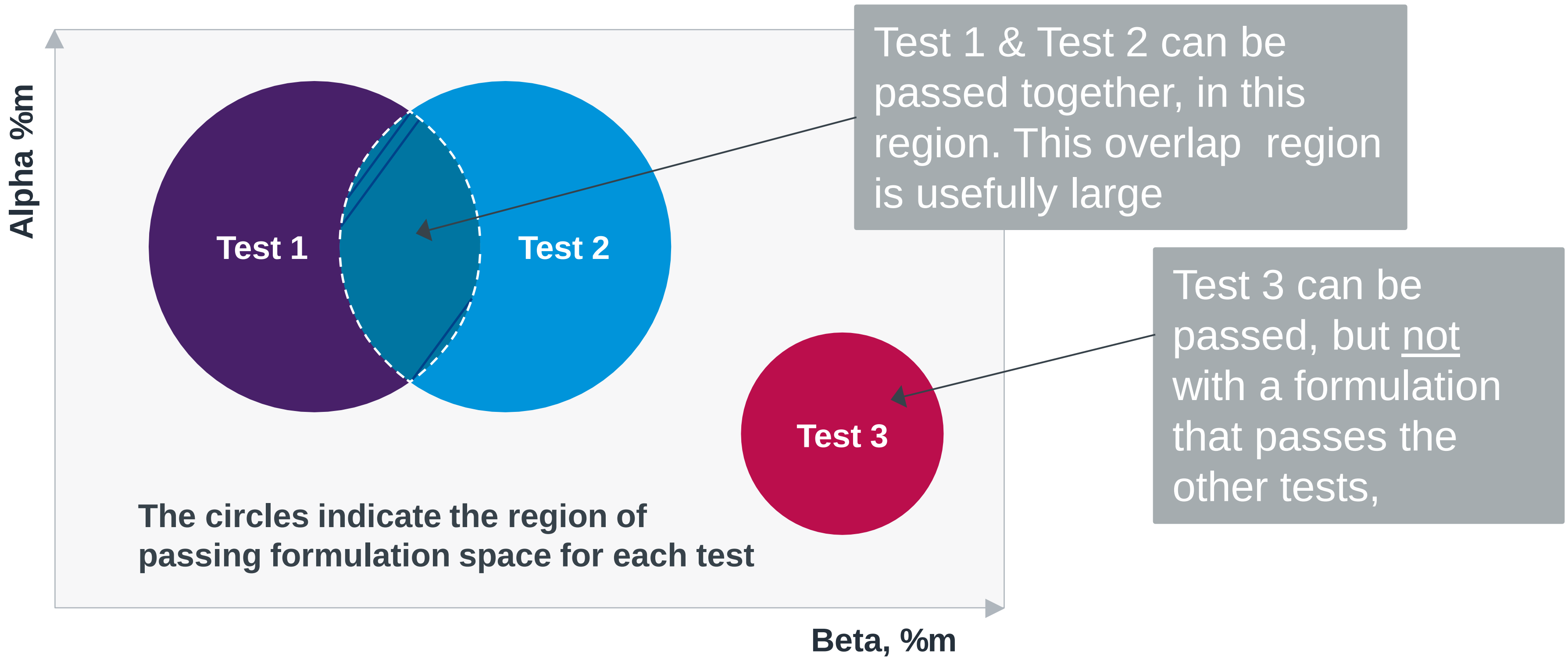
Example of a formulation problem (2)

Plot of the passing regions of “Formulation Space” for each test. Using components Alpha and Beta



There is no formulation above that can pass all three tests together. This is a problem for this project.

Example of a formulation problem (3)

Later, in the same development

- A colleague has discovered that the response of Test 3 to Alpha and Beta can be altered beneficially by adding 1% of a new component, Gamma, to the system
- We therefore add 1% of Gamma to the system and re-explore the formulation space of each test across differing treat rates of Alpha and Beta
- We see what happens in this case, in the next slide

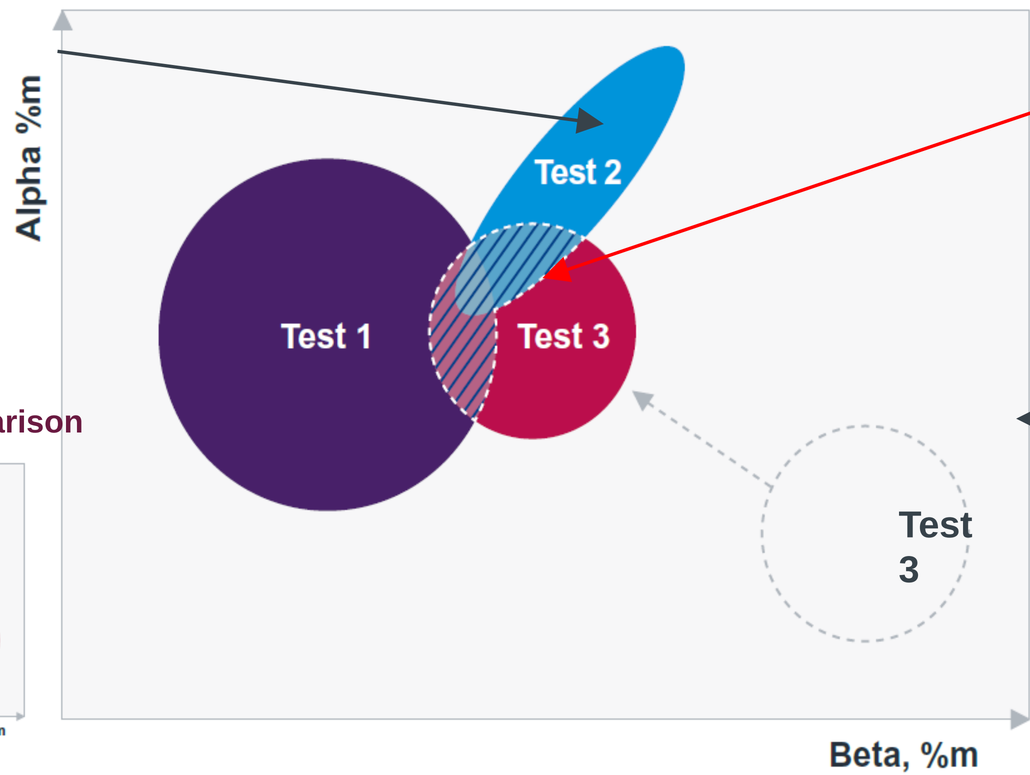
Example of a formulation problem (4)

Plot of the passing regions of “Formulation Space” for each test.
Using components Alpha, Beta, with 1%m Gamma

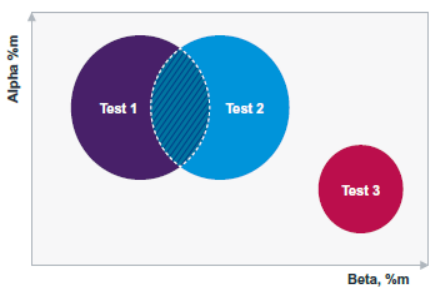
3. However, the use of Gamma has also changed the response shape of Test 2

2. A region of formulation space can pass all three tests

1. The passing region of Test 3 has now moved to a more useful place



Previous case, for comparison



4. Due to the changed response shape of Test 2, the formulation window above is, in practice, too small to represent a robust solution, given test scatter.

Example of a formulation problem (5): conclusions

The concept of formulation balance

- In this example, we have seen how the addition of a component can have a positive effect on one test, but also a negative effect on another
- This is a very common occurrence in crankcase formulations and the skill of the formulator is to set an appropriate balance between all the various opposing effects
- In a crankcase development, where there can be 50 tests and 10 or more additive components, the task of achieving balance is not always straightforward
- In the next slide we outline some examples of opposing component effects in crankcase formulations

Formulation balance: examples

Potential Positive Effects		Potential Negative Effects
• Soot-induced oil thickening • Sludge control		• Fuel economy, • Fluoroelastomer seals
• Acid control • Piston cleanliness • Anti-rust	Dispersant Mixture	• Particulate filter blocking • Wear • Oil haze
• Fuel economy	Overbased Detergent Mixture	• Package instability • Oil haze
• Elastomer seals • Additive solubility • Oxidation resistance	Organic Friction Modifier	• Valve train wear
• Film thickness (viscosity contribution) • Fuel economy	Base Stock Ester	• Viscosity loss on shear • Piston cleanliness
	Viscosity Modifier	

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Formulation aspects: base stock

Typically two or more base stocks of differing viscosities are blended in a mixture to meet targets as outlined below

Viscometrics

- Viscosity at cold temperatures (cold cranking simulator (CCS) test)
- As required by viscosity grade, e.g. 0W-X vs. 5W-X

Volatility

- Lower W-grades have higher volatilities for a given base stock type, e.g. 0W-30 >5W-30
- Volatility can be reduced by including low volatility base stock types, e.g. PAO (expensive)

Engine- and bench test performance

- Base stock choice can be influenced also by performance requirements:
    - Engine tests (typically in areas of piston cleanliness, engine sludge and in soot thickening)
    - Seals tests (can be poor in highly non-polar base stocks)

Formulation aspects: viscosity modifier

Viscosity modifier (VM) choice depends on a balance of factors:

HTHS viscosity	Contribution to High Temperature High Shear (HTHS) viscosity for a given SAE grade must be sufficient
Shear stability	Viscosity reduction upon mechanical shear testing must not be greater than specification limits
Volatility	VM diluent oil contributes to finished oil volatility. Group III diluent provides lower volatility than Group I
Cold temperature	Choice of VM can affect wax drop out and cold flow in used oils
Engine performance	Engine cleanliness can be impacted negatively by VM Fuel economy can benefit from appropriate VM
Customer logistics	Often, customer logistics impact choice of VM

Formulation aspects: detergent system (a)

Range of detergents	A wide range of detergents is available, including • Salicylates, phenates, sulphonates • Highly overbased and neutral components • Magnesium or calcium metal
Use of mixtures	Typically, a mixture of detergents is used, e.g. overbased and neutral components) so as to provide a balanced mixture of attributes

Alkalinity reserve	• Overbased detergents contribute strongly to alkalinity reserve, as measured by Total Base Number (TBN)
	• The overbased component mixture is chosen such that
	    • The reserve of alkalinity is adequate in field performance
	    • Meet any specification criteria for minimum TBN either for new oil or for oil after specific engine tests

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Formulation aspects: detergent system (b)

Ash contribution	• Ash contribution is the "other side of the coin" to alkalinity reserve • Some engines, and particulate filters respond badly to ash. Thus some specifications place maximum allowed values on ash, e.g. 0.8%m • Thus the detergent system is balanced such that the oil meets any ash constraints
Other aspects	• The levels of neutral detergents are chosen to provide adequate detergency for, e.g. piston cleanliness • The choice of detergent metal can influence wear performance
Stability	• Overbased detergents can drop out of suspension, causing haze or deposits in detergent inhibitor (DI) or oil • Issues can arise depending on the mix of other components and on polarity of base stock (PAO can be poor)

Formulation aspects: dispersant system

The levels and types of dispersant in the formulation are chosen to provide a balance of properties as outlined here.

Positive attributes

The levels & selection of components are chosen to provide tailored performance in the areas of
- Control of soot-induced oil thickening
- Black sludge control, filter plugging
- Piston and engine cleanliness in key tests

Other aspects to consider

- Oil viscosity at cold temperatures is increased by high molecular weight dispersant
- Fluoroelastomer performance can be degraded by nitrogen containing dispersants
  - May mitigate via use of seals fix
- Fuel economy can be degraded at higher dispersant levels
  - Need to adjust other formulation aspects to compensate for this

Formulation aspects: other components (a)

Anti-oxidants	• Aminic or phenolic anti-oxidants are often included
	  • Tests can respond differently to aminic and phenolic types so a balanced mixture may be used
	• Other component types also provide anti-oxidancy
	  • ZDDPs: highly potent & highly cost effective
	  • Metal and/or sulphur containing friction modifiers (FM)

ZDDPs	• Universally used anti-wear and anti-oxidant additives • Secondary ZDDPs commonly used, due to their high level of wear protection • Primary ZDDPs are also available and can find more specialist application due to their higher stability & longevity • ZDDPs contribute phosphorus, ash and sulphur to the oil (harmful to emission control systems) and so their level is often restricted in specifications. • e.g. ACEA C3-16 (0.09%m max) or ACEA E6-16 (0.08%m max)

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Formulation aspects: other components (b)

Friction modifiers

- May be metal containing (e.g. molybdenum) or organic
- Are surface active components so can affect anti-wear performance
- Organic FMs can cause stability issues in the detergent inhibitor (DI) package or finished oil
  - May require DI to be delivered as two packages

Others include

- Corrosion inhibitors, demulsifiers
  - Beware of wear issues (due to their surface activity)
- Seals fixes
- Pour point depressants
  - Component may be mandated by customer
  - Treat rate depends on base stock and on rest of formulation
  - Industry codes generally allow read across from one component to another

Formulation aspects: stability/harms

Stability

- Once a formulation is defined, it is essential to check that the package and oil are stable
- Early indications of instability are haze or formation of visible layers
- Detergent overbased material can drop out, depending on its chemical environment
    - Care needed with highly non-polar base stocks
- Friction modifiers can induce stability issues
- Requires storage stability testing, typically to 12 weeks

Harms testing

- It is prudent also to check early for any unexpected effects via testing in laboratory tests, e.g.
- Filterability
- Foaming
- Oxidation tests
- Cold temperature viscometrics
- Seals & corrosion tests

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Formulation for key engine tests

Some examples of engine tests in current ACEA & API specifications and the key oil properties that they probe are listed below

Passenger Car Motor Oil

Property	Relevant test
Black sludge	M271SL, Seq. VG*
Soot induced oil thickening	DV6
Piston cleanliness, ring sticking	VW TDI, EP6
Oxidative oil thickening	Seq. IIIG*
Valve train wear	OM646LA, Seq. IIIG*, Seq. IVA*
Fuel economy	M111FE, Seq. VID*
Bearing corrosion	Seq. VIII,

Heavy-duty Diesel Oil

Property	Relevant test
Soot induced oil thickening	Mack T8E, Mack T-11
Filter plugging	Cummins ISM
Piston cleanliness, ring sticking	OM501LA, Cat C13, Cat 1N
Valve train wear	Cummins ISM, Cummins ISB, RFWT, OM646LA
Ring/liner wear	OM501LA, Mack T12
Aeration	EOAT, COAT
Oxidative oil thickening	Volvo T-13
Bearing corrosion	Mack T12

Further testing requirements, not listed here, may include OEM requirements, either in-house OEM engine tests or OEM field trials

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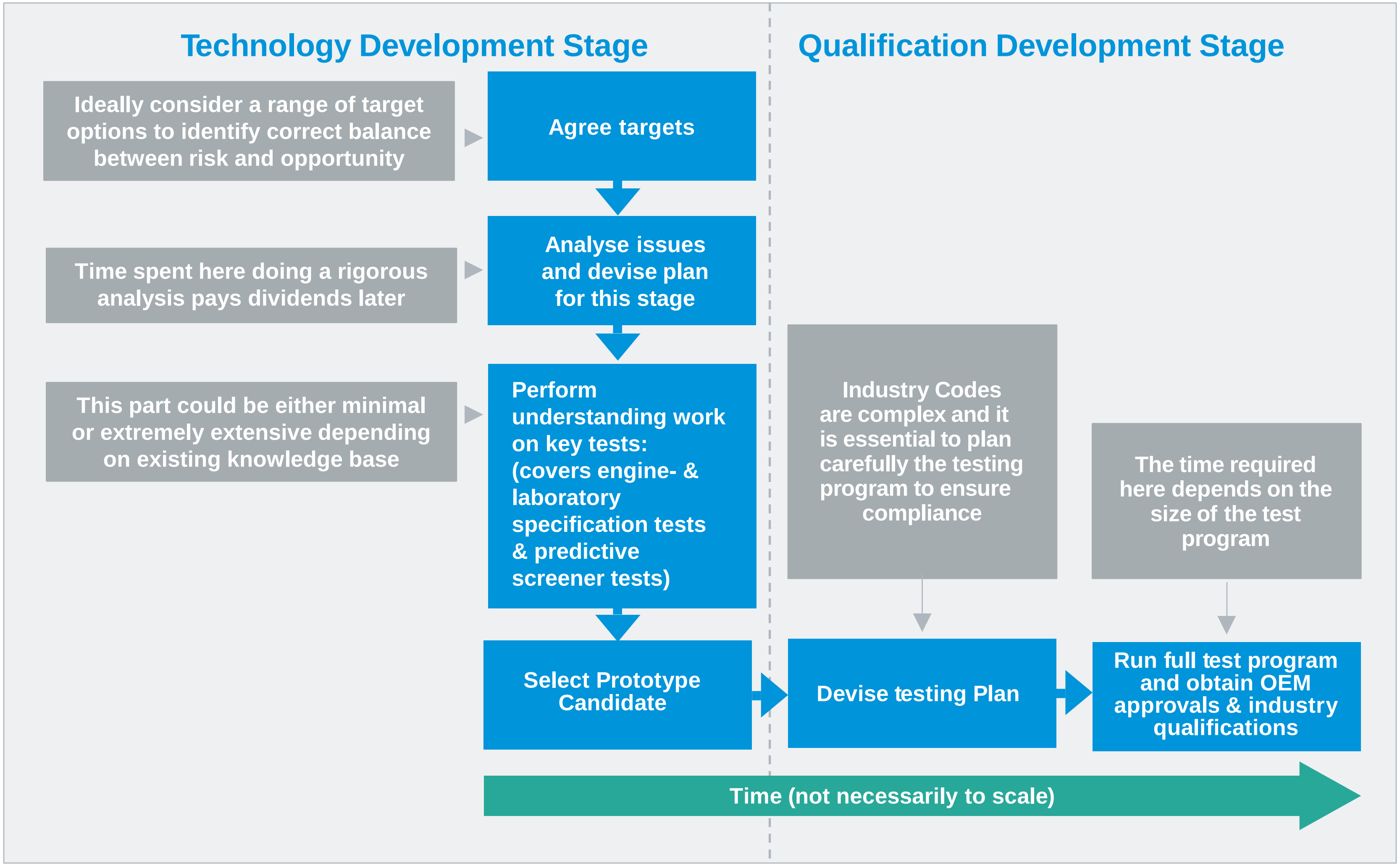
Stages in a product development program



Product development program: time & cost

For a product development, the elapsed time to completion and testing costs can vary widely depending on its complexity & challenge. Contrasting realistic examples are outlined below:

Lower-end complexity & challenge	• Add a single ACEA specification to an existing product, e.g. ACEA C3-16
	• We assume no need for technology development work
	    • Testing cost (= once-through costs): 400k USD
	    • Elapsed time to complete: 2-3 months

Higher-end complexity and challenge	• Develop product meeting combinations of demanding OEM specifications (Passenger Car or Heavy Duty Diesel) with extensive testing requirements (as well as ACEA specifications)
	• Let's assume, for illustration purposes, that considerable technology development work is required
	    • Total project cost: > 5 million USD
	    • Elapsed time to complete: > 3 years

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Codes of practice framework

Codes of practice	• Qualification testing for Industry specifications, e.g. API SN, ACEA C3-16 must conform to industry codes of practice • These Codes of Practice have been put in place to assure the quality of the tested product • The Codes for US and European specifications follow the same principles, although differing in some details

Principles include	• All test work should be visible to the customer
	  • All engine tests are registered with a third party
	  • Mandatory requirement to provide a defined data package on request
	• Any read-across of results (that promotes efficiency of testing) should not compromise the quality of the product
	  • Read across rules put in place by the Industry that are derived from data analysis
	  • In essence, the most severe case is tested and read across allowed only to less demanding cases

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Qualification testing: requirement for planning

- The design of any qualification testing program is impacted considerably by the requirements of the codes of practice.
  - As noted earlier, it is essential to consider carefully these requirements at the program planning stage so as to ensure that the test program is fully compliant with the codes
- Specifically, the codes impose specific constraints on design aspects of the test program, including:

Number and content of detergent inhibitor (DI) modifications within a program

Read-across of results from one viscosity grade to another

Read-across of results from one base stock to another

Changes in viscosity modifier levels used within a program

Changes of ratio of base stocks used within a program

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Summary

Crankcase formulation: what's it all about?

- Satisfying a huge number technical requirements which often oppose each other.
- Recognising the concept of formulation balance
- Understanding the positive and negative impacts of the individual components
- Planning very carefully the technology development scoping work as well as the detail of the qualification test work
- Investing potentially very large engine test budgets where programs are complex and technically challenging
- Adhering to industry codes of practice

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