

# **AMERICAN CHEMISTRY COUNCIL PRODUCT APPROVAL CODE OF PRACTICE**

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## **APPENDIX H**

### **GUIDELINES FOR MINOR FORMULATION MODIFICATIONS**

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### GUIDELINES FOR MINOR FORMULATION MODIFICATIONS

#### **Introduction**

Minor formulation modifications are part of the American Chemistry Council Code for testing engine oils. These guidelines were developed based on criteria set by a work group of industry formulators. The guidelines are based on fundamental knowledge of the performance of engine oils in each test type. They relate to industry need and have been verified by industry data. No guideline is driven by individual company need.

#### **Purpose**

For all engine tests accepted into the Code of Practice, minor formulation modifications can be applied. This appendix outlines the allowable modifications which can be made during the development of a Core Data Set (Tab 1).

During the development of the Core Data Set, Minor Formulation Modification Guidelines and API Guidelines for SAE Viscosity-Grade Engine Testing (API 1509 Appendix F) may be used. Additionally, for a matrix approach, API Base Oil Interchangeability Guidelines (API 1509 Appendix E) may also be used.

#### **General Guidelines**

Guidelines for minor formulation modifications allow the formulator to make small adjustments in the candidate formulation during the conduct of a test program so that a failed test(s) does not force discarding passing results for previously run test types. Such minor modifications are made with the intent that they result in a discernible improvement in performance. Minor formulation modifications made during the conduct of a Program are based on fundamental formulation knowledge and can include but are not limited to those modifications described in "Guidelines for Specific Engine tests."

If minor formulation modifications are used during the conduct of an engine test program, such minor modifications are permitted with the expectation that the final formulation contain all modifications and will pass all the engine and chemical & physical tests required by the performance claim. Supporting data will be required to ensure that minor modifications will not deteriorate performance in tests previously passed.

All minor modifications and support data will be disclosed to and agreed to by the customer and included in the Candidate Data Package (Appendix E).

The General Guidelines for minor modifications apply to all of the tests accepted into the ACC Code of Practice. Specific guidelines are provided for the following engine test Sequences IIIF, IIIG, IIIH, IIIH60, IIIH70, IVA, IVB, VG, VH, VID, VIE, VIF, VIII, IX, and X are listed in the section titled "Guidelines for Specific Engine Tests".

#### **Guidelines for Specific Engine Tests**

The numbered guidelines listed here are applicable only to Sequence IIIF, IIIG, IIIH, IIIH60, IIIH70, IVA, IVB, VG, VH, VID, VIE, VIF, VIII, and IX engine tests. Guideline 11 must be consulted when applying these guidelines to the Sequence IX test as indicated by footnote 1 in this section. Specific tests have been included in these guidelines based on a thorough

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review by the Minor Formulation Modification Working Group and acceptance by the Petroleum Additives Product Approval Protocol Task Group. These tests have been judged to respond either beneficially or without harm to formulation changes allowed by the numbered guidelines. This judgment is based on collective internal company data, previous generation tests and on basic formulation knowledge.

No guideline is driven by individual company data.

New tests may be considered for inclusion in these Specific Guidelines if:

- The test has been added to a new or revised API Category
- The engine test has been accepted into the Code of

Practice Engine tests may be considered for removal from these

Specific guidelines if:

- The test becomes obsolete or is removed from the COP
- Changes in test hardware or procedures indicate that the engine test no longer responds to changes in additive chemistry

Common industry terminology is used to describe ingredients in the candidate formulation impacted by the numbered guidelines.

Additive treatment levels in the following guidelines are in percent mass. Major components are those included as part of the performance additive package at a treatment level of >1.0% in the formulation to be tested. Decrease in the treatment level of components of the performance additive package other than for rebalances (Guidelines 5 and 6), is not allowed. All modifications are relative except those that are noted as absolute. Definitions for Level 1 and Level 2 support are found in Tab 1.

1. An increase in the treatment level of the performance additive package, exclusive of viscosity modifier and pour point depressant, is acceptable.
  - a)  $\leq 20\%$  with Level 1 support<sup>1</sup>.
  - b)  $> 20\%$  to  $\leq 30\%$  with Level 2 support.
2. An increase in the treatment level of a single component of the performance additive package present at greater than 1.0% (major component) in the formulation to be tested is acceptable:
  - a)  $\leq 20\%$  with Level 1 support<sup>4</sup>.
  - b)  $> 20\%$  to  $\leq 30\%$  with Level 2 support.
3. An increase in the treatment level of a single component of the performance additive package present at 1.0% or less in the formulation to be tested is acceptable:
  - a)  $\leq 0.3\%$  to  $\leq 100\%$  with Level 1 support<sup>4</sup>;  $> 100\%$  to  $200\%$  (maximum 0.6% in formulation to be tested) with Level 2 support.
  - b)  $> 0.3\%$  to  $\leq 0.6\%$  to  $\leq 50\%$  with Level 1 support<sup>4</sup>;  $> 50\%$  to  $100\%$  with Level 2 support.
  - c)  $> 0.6\%$  to  $\leq 1\%$  to  $\leq 30\%$  with Level 1 support<sup>4</sup>;  $> 30\%$  to  $100\%$  (maximum 1.3% in formulation to be tested) with Level 2 support).
4. With Level 2 support, one new component not present in the original formulation may be added. The new component may not exceed 10% of the total performance additive package (original package plus added component).

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<sup>1</sup> See guideline 11 for the Sequence IX test

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5. Variations in zinc dithiophosphate (ZDP) type and treatment level are acceptable changes with appropriate Level 1 or Level 2 support.
    - a) Rebalance among zinc dithiophosphate (ZDP) is allowed while maintaining a constant formulation phosphorus level with Level 2 Support. This may include introduction of a new ZDP; only one new ZDP introduction is allowed. Such a rebalance counts as one minor modification. Only one ZDP rebalance is allowed.
    - b) An increase in treatment level of zinc dithiophosphate (ZDP), in a formulation where the phosphorus level from ZDP is greater than 0.04%, up to a maximum of 0.12% phosphorus from ZDP is acceptable with Level 2 support for the Sequence VID, VIE and/or VIF and Level 1 support for all other engine tests. For increases above 0.12% P from ZDP, Level 2 support is required for all engine tests. Alternatively, Guideline H2 or H3 could be used if applicable.
  6. A rebalance of metallic detergents is acceptable with Level 2 support provided that the sulfated ash remains constant and the metallic detergent soap is not decreased. For any individual detergent soap type, the increase in soap level is limited to 30% maximum. Only one detergent rebalance is allowed.

The detergent rebalance may be effected simultaneously with the addition of a new metallic detergent component in line with the requirements of Guideline 4. In the specific instance where the soap and metal type of the new component are already present in the formulation, the rebalance counts as one modification. In other cases, the simultaneous rebalance/addition counts as two minor modifications.

7. There is a limit to the number of minor modifications allowed during the conduct of a Core Program (see below). This limit applies to Guidelines 1 through 6 only. For Guidelines 1 through 4, if the same minor modification guideline is applied more than once and the sum falls within the guideline parameters, this is considered to be only one minor modification. The sum of all modifications shall not result in an increase in treatment level of any major component of the performance additive package of greater than 30%.
  - a) When using a non-matrix approach, no more than three minor modifications, made either individually or simultaneously, may be incorporated in the core data set.
  - b) When using a matrix approach, a maximum of four minor modifications may be used.
8. Base stock ratio and viscosity modifier treatment level (not type) are acceptable changes with Level 1 support.
  - a) A 15% absolute change in base stock ratio within the same base stock slate (+ or - 15% compared to the wt. % of the base oil blend) is allowed with Level 1 support. This change can include the addition of a new base stock cut that is part of the original base stock slate.
  - b) If a new base stock is added and is in a different base stock slate and that slate is either API Group I, Group II, Group III or Group IV the change is limited to a maximum of 10% of the formulation (the finished blend of base stocks and additives).
  - c) Viscosity modifier (either dispersant or non-dispersant type) treatment level may change no more than 15% relative to its treat rate.  
["Type" means a specific molecular structure with a specific shear stability characterized by a specific trade name, stock or code number.]
  - d) Base stock ratio and/or viscosity modifier level changes greater than those cited

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above in 8a, 8b or 8c are allowed with Level 1 support as permitted by the API BOI/VGRA guidelines as defined in API 1509 for a given test.

9. Variations in pour point depressant and/or foam inhibitor type or treatment level are acceptable changes with Level 1 support. When changing foam inhibitor type or treatment level in the Caterpillar engine Oil Aeration Test (COAT), Level 1 support alone is not adequate; fundamental formulation knowledge support must exist to ensure performance is not deteriorated in this test.
10. The performance additive package commercialized for sale must include all permitted minor modifications in accord with Guideline 7. The package plus any other minor modifications made under Guidelines 8 and 9 define the final formulation.
11. Guidelines 1 through 6 can be used with the Sequence IX test, however, all modifications which involve a metallic detergent (e.g. performance additive package treat rate increase, metallic detergent increase, metallic detergent rebalance, or new metallic detergent introduction) requires Level 2 support for the Sequence IX.

## ILLUSTRATIONS OF GUIDELINE USAGE

### Introduction

The following illustrations are only examples, and do not preclude other ways, of application of minor formulation modification guidelines. Where multiple illustrations are used for a single guideline they are represented by a hyphenated number, the first part of which refers to the specific guideline number.

### Illustration 0 Matrix Core Data Set

As an alternative to the use of a single base stock slate for the generation of the Core Data Set to support an ACC Program, a matrix approach may be used. A Matrix Core Data Set uses the API interchange guidelines in that a test (or tests) may be run in any applicable base oil combination. Not all base stock slates or base oil combinations used in a Matrix Core may be qualified to carry the final API category claim.

For example, an API SM matrix to cover base stock slate D might look like this\*:

Base Oil (Group)	A (II)	B (II)	C (III)	D (II)
Viscosity Grade	10W-40	10W-40	5W-30	10W-40
Base Oil Saturates	91	98	99	99
Base Oil Sulfur	0.005	0.000	0.000	0.000
Base Oil Vis @ 100C, cSt	5.8	6.0	5.6	6.2
Sequence III G	-	-	-	Run
Sequence IV A	-	Run	-	BOI
Sequence V G	Run	-	-	BOI
Sequence VIII	-	-	Run	VGRA/BOI

API SM is allowed in base stock slate D. In order to qualify the other base stock slates, a Sequence III G, as well as possibly other engine tests, would be required in that base stock slate or base oil combination per the API Base Oil Interchangeability Guidelines, the API Guidelines for SAE Viscosity- Grade Engine Testing and the API Guidelines for Use of a Single Technology Matrix.

For example, an API SM/EC matrix to cover base stock slate H might look like this\*:

Base Oil (Group)	E (II)	F (II)	G (III)	H (II)
Viscosity Grade	5W-30	5W-30	5W-20	5W-30
Base Oil Saturates	96	98	99	99
Base Oil Sulfur	0.001	0.000	0.000	0.000
Base Oil Vis @ 100C, cSt	4.2	4.1	4.2	4.3
Finished Oil CCS, cP	6100	5900	5780	5800
Finished Oil HTHS, cP	3.0	2.9	2.6	2.9
Sequence III G	-	-	-	Run
Sequence III GA	-	-	-	Run
Sequence IV A	-	Run	-	BOI
Sequence V G	Run	-	-	BOI
Sequence VIII	-	-	Run	VGRA/BOI

API SM/ Energy Conserving is allowed for Base stock slate H.

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For example, an API CJ-4/SM 15W-40 matrix might look like this\*:

Base Oil (Group)	I (I)	J (I)	K (II)	L (II)
Base Oil Saturates	78	85	90	97
Base Oil Sulfur	0.3	0.1	0.001	0.000
Base Oil Vis @ 100C, cSt	5.2	5.3	5.3	5.5
Base Oil Viscosity Index	100	101	106	110
Sequence IIIG	Run	Run	Run	Run
Sequence IVA	Run	BOI	BOI	BOI
Sequence VG	Run	BOI	BOI	BOI
Sequence VIII	BOI	Run	BOI	BOI
Caterpillar 1N	BOI	BOI	BOI	Run
Caterpillar C13	Run	BOI	BOI	Run
RFWT	BOI	Run	BOI	BOI
Cummings ISM & ISB	Run	BOI	BOI	BOI
Mack T-11	Run	BOI	BOI	BOI
Mack T-12	Run	BOI	BOI	BOI

API CJ-4 / SM is allowed for all four base stock slates above.

Since API BOI and VGRA Guidelines are subject to change, it is prudent to review the most current version of API Publication 1509, Appendix E and F, before initiating a Matrix Approach Core Data Set Test Program.

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### **Illustration 1(a) For Guideline 1(a)**

A performance additive package is used in the candidate at 10.0% mass. During the development of the Core Data Set, the candidate passes one or more of the required tests; however, it is necessary to increase the treatment level of the performance additive package to 12.0% mass to pass the remaining tests. This minor modification requires Level 1 support.

### **Illustration 1(b) For Guideline 1(b)**

Conditions exist as outlined in Illustration 1(a) but during the development of the Core Data Set, the treatment level of the performance additive package is increased to 13.0% mass to pass the remaining tests. This minor modification requires Level 2 support demonstrating no harm at the 13.0% mass treatment level in the test(s) run at 10.0% mass.

### **Illustration 2(a) For Guideline 2(a)**

A component is present in the formulation to be tested at 2.0% mass. During the development of the Core Data Set, it is necessary to increase the component to 2.4% mass in the formulation to be tested to pass the remaining tests. This minor modification requires Level 1 support.

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### **Illustration 2(b) For Guideline 2(b)**

A component is present in the formulation to be tested at 2.0% mass. During the development of the Core Data Set, it is necessary to increase the component to 2.5% mass in the formulation to be tested to pass the remaining tests. This minor modification requires Level 2 support demonstrating no harm for the 2.5% mass treatment level of the component in the tests run with 2.0% mass treatment.

### **Illustration 3(a) For Guideline 3(a)**

A component is present in the formulation to be tested at 0.2% mass. During the development of the Core Data Set, it is necessary to increase the component to 0.5% mass in the formulation to be tested to pass the remaining tests. This minor modification requires Level 2 support demonstrating no harm for the 0.5% mass treatment level of the component in the tests run with 0.2% mass treatment.

### **Illustration 3(b) For Guideline 3(b)**

A component is present in the formulation to be tested at 0.4% mass. During the development of the Core Data Set, it is necessary to increase the component to 0.6% mass in the formulation to be tested to pass the remaining tests. This minor modification requires Level 1 support.

### **Illustration 3(c) For Guideline 3(c)**

A component is present in the formulation to be tested at 0.7% mass. During the development of the Core Data Set, it is necessary to increase the component to pass the remaining tests. A 100% increase in the treatment level of the component is desired. However, the guideline limits the formulation to a maximum of 1.3% mass of the component in the formulation to be tested. The minor modification requires Level 2 support demonstrating no harm for the 1.3% mass treatment level of the component in the tests run with 0.7% mass treatment.

### **Illustration 4 For Guideline 4**

A performance additive package is used in the candidate at 10% mass. During the development of the Core Data Set, it is necessary to add a new component not present in the original candidate to pass the remaining tests. Addition of this component is limited to 10% of the performance additive package (original package plus added component). This minor modification requires Level 2 support demonstrating no harm for the new component in the tests run prior to its addition.

### **Illustration 5-1 For Guideline 5a**

The performance additive package contains a 50/50 mixture of ZDP A and ZDP B. During the development of the Core Data set, it is necessary to increase the treatment level of ZDP B by 25% (25% ZDP A/75% ZDP B) to pass the remaining tests. This maintains a constant formulation phosphorous level. This minor modification requires Level 2 support.



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### **Illustration 5-2 For Guideline 5a**

The performance additive package contains 100% of ZDP A. During the development of the Core Data Set, it is necessary to add ZDP B up to 25% to pass the remaining tests. This minor modification (75% ZDP A/25% ZDP B) maintains a constant formulation phosphorous level. This requires Level 2 support demonstrating no harm in those tests run with 100% ZDP A.

### **Illustration 6-1 For Guideline 6**

The performance package contains a mixture of calcium sulfonate detergents. During the development of the Core Data Set, it is found necessary to increase the sulfonate detergent soap level by 30%. This is achieved by adjusting the ratio of the two calcium sulfonate detergent components to attain the required increase in detergent soap while maintaining a constant sulfated ash level. This minor modification requires Level 2 support demonstrating no harm for the performance package containing the adjusted concentrations of the metallic detergents in those tests run with the metallic detergents in the original concentrations.

### **Illustration 6-2 For Guideline 6**

The performance package contains a mixture of magnesium and calcium sulfonate detergents. During the development of the Core Data Set, it is found necessary to increase the concentration of the magnesium sulfonate detergent component by 30%, e.g., Mg content increased from 0.05% mass to 0.065% mass in the finished oil, without simultaneously increasing the sulfonate detergent soap level. The concentrations are adjusted so that overall there is no change in the sulfated ash and sulfonate soap levels in the finished oil. This minor modification requires Level 2 support demonstrating no harm for the performance package containing the adjusted concentrations of the metallic detergents in those tests run with the metallic detergents in the original concentrations.

### **Illustration 6-3 For Guideline 6**

The performance package contains a mixture of calcium sulfonate and calcium phenate detergents. During the development of the Core Data Set, it is found necessary to increase the sulfonate detergent soap level by 30%. However, only one calcium sulfonate detergent component (A) is present in the formulation. To maintain a constant sulfated ash content it is necessary to adjust the concentration of the calcium sulfonate (A) and introduce a new lower TBN calcium sulfonate component (B). Since detergent component B is of the same soap and metal type as a detergent already in the formulation, i.e., detergent component A, then the change counts as one minor formulation change and requires Level 2 support demonstrating no harm for the performance package containing the adjusted concentrations of the metallic detergents in those tests run with the metallic detergents in the original concentrations.

### **Illustration 7-1 For Guideline 7**

During the development of the Core Data Set, it is necessary to increase the total performance additive package by 25% (Guideline 1(b)), rebalance the ZDP (Guideline 5), and add a new component not present in the original candidate (Guideline 4). These minor modifications require Level 2 support in accord with the individual guidelines.

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### **Illustration 7-2 For Guideline 7**

Conditions exist as outlined in Illustration 7-1 but during the development of the Core Data Set it is determined that a rebalance of metallic detergents is necessary (Guideline 6). Such a minor modification exceeds the three that are allowed. Some of the tests previously run must be repeated to assure that only three minor modifications are used from the start to completion of the Core Data Set. These minor modifications require Level 2 support in accord with the individual guidelines.

### **Illustration 7-3 For Guideline 7**

During the development of the Core Data Set, it is necessary to increase the treatment level of a single component present in the formulation to be tested at 0.4% mass by 50% (Guideline 3(b)) and increase the treatment level of a second component present in the formulation to be tested at 0.8% by 30% (Guideline 3(c)). It is also necessary to increase the treatment level of the total performance additive package by 15%. While each of these minor modifications require Level 1 support, the aggregate of the modifications results in increases in the individual components exceeding Level 1 limitations. Under such conditions, Level 2 support is required.

### **Illustration 7-4 For Guideline 7**

During the development of the Core Data Set it is necessary to increase the treatment level of the total performance additive package by 15% (Guideline 1(a)), then increase the treatment level of the performance additive package again by 15% relative to the original level. The total increase in the treatment level of the additive package is now 30% (15% + 15%), done in two steps (Guideline 1(b)). Overall, there is only one minor modification. While each individual increase in treatment level of the performance additive package requires Level 1 support, the overall increase requires Level 2 support.

### **Illustration 7-5 For Guideline 7**

During the development of the Core Data Set it is necessary to increase the treat level of a single component present at 2.0% mass by 10% (Guideline 2(a)), resulting in a treat level of 2.2% mass. The treat level of this component is then increased a second time by 15% (Guideline 2(a)), resulting in treat level of 2.53% mass and then a third time by 5% mass (Guideline 2(a)), resulting in a treat level of 2.66% mass. These increases in the treat level of the same component are considered only one minor modification. While each of these increases requires level 1 support, the sum of the first two increases exceeds Level 1 limitations and Level 2 support is required. The aggregate of the three increases totaling a 33% increase, results in the individual component exceeding Level 2 limitations of 30% maximum, therefore additional engine testing would be required.

### **Illustration 8-1 For Guideline 8**

The formulation contains two base stocks A and B. During the development of the Core Data Set, it is determined that it is necessary to change concentration of base stock A in the base stock combination by 15% absolute to maintain the same viscosity characteristics. Base stock B is correspondingly adjusted.

Absolute percentages are calculated by first normalizing the base stock portion of the formulation to 100%. For example, a formulation containing 60% base stock A and 20% base stock B, together with 20% of other components, such as additives and viscosity modifier, has absolute percentages of base stocks A and B of 75% and 25% respectively. A maximum

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allowable reduction of 15% absolute in base stock A utilizing Guideline 8 would produce absolute percentages of 60% and 40% for A and B respectively, corresponding to final formulation (relative) percentages of 48% A, 32% B, and 20% of other components.

### **Illustration 8-2 For Guideline 8**

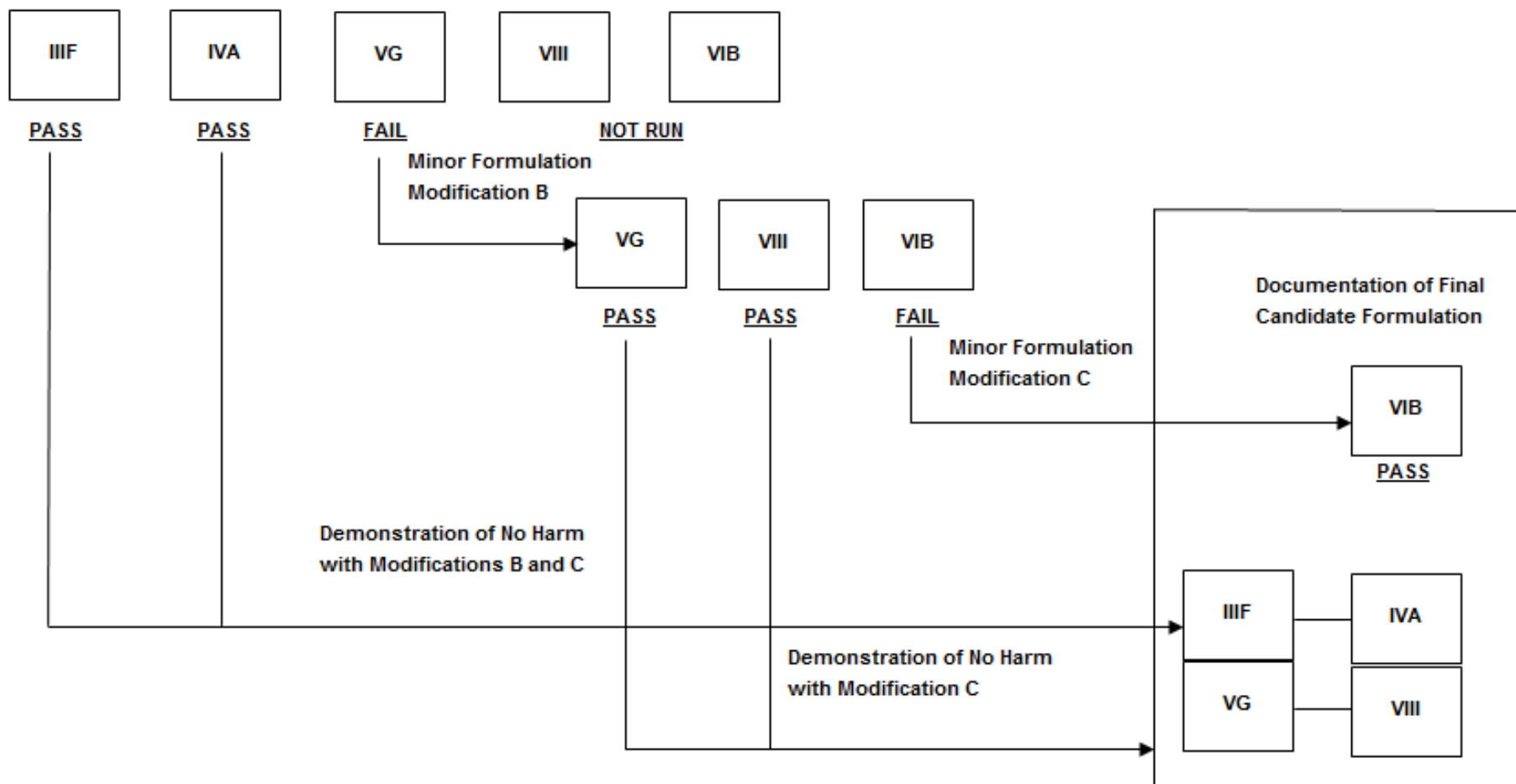
The formulation contains a single base stock A. During the development of the Core Data Set, it is determined that to maintain the same viscosity characteristics it is necessary to change 15% absolute of the base stock by replacing base stock A with base stock B from the same slate. The treatment level of viscosity modifier remains unchanged. This minor modification requires Level 1 support.

### **Illustration 9 For Guideline 9**

During the development of the Core Data Set, it is determined that use of a different foam inhibitor is required. This minor modification requires Level 1 support.

## ILLUSTRATION OF APPLICATION OF MINOR FORMULATION MODIFICATIONS

Original Formulation (Coded as Modification A; See Appendix B, Page 4)



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