# ANALYSIS OF ALTERNATIVES

## Non-confidential Report

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| **Legal name of applicant(s):** | *Indestructible Paint Limited* |
| **Submitted by:** | *Indestructible Paint Limited* |
| **Substance:** | *Pentazinc chromate octahydroxide (zinc tetrahydroxide chromate); CAS 49663-84-5* |
| **Use title:** | *Use of Pentazinc chromate octahydroxide in stoved epoxy primer for corrosion protection of aircraft engine components in aerospace and aeroderivative applications* |
| **Use number:** | *1 and 2* |

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## LIST OF ABBREVIATIONS

| | |
|---|---|
| AA3003 | Aluminium alloy with specific composition |
| AAR | Aviation Accident Report |
| Acute Tox. | Acute Toxicity |
| AfA | Application for Authorisation |
| Al | Aluminium |
| AMMTIAC | Advanced Materials, Manufacturing, and Testing Information Analysis Center |
| AoA | Analysis of Alternatives |
| Aquatic Acute | Hazardous to the aquatic environment |
| Aquatic Chronic | Hazardous to the aquatic environment |
| ASTM | American Society for Testing and Materials |
| B-737 | Boeing 737 |
| BS | British Standards |
| °C | Celsius |
| Carc. | Carcinogenicity |
| CAS | A unique numerical identifier assigned by Chemical Abstracts Service (CAS number) |
| CLP | Classification, Labelling and Packaging |
| COLM | Corrosion on Light Metals |
| Cr | Chromium |
| Cr(VI) | Hexavalent Chromium |
| CS-25 | Certification Specification for Large Aeroplanes |
| CS-E | Certification Specification for Engines |
| CSR | Chemical Safety Report |
| Cu | Copper |
| EASA | European Aviation Safety Agency |
| EC | A unique numerical identifier of the European Community (EC number) |
| ECHA | European Chemicals Agency |
| e.g. | Exempli gratia, for example |
| EHS | Environmental Health and Safety |
| EN | European Norm |
| EU | European Union |
| Eye Dam. | Serious eye damage |
| FAA | Federal Aviation Administration |
| Fe | Iron |
| GCCA | Global Chromates Consortium for Aerospace |
| GHS | Global Harmonised System |
| h | Hour |
| HITEA | Highly Innovative Technology Enablers for Aerospace |
| IAEG | International Aerospace Environmental Group |
| IDP Ltd. | Indestructible Paint Limited |

| | |
|---|---|
| ISO | International Organisation for Standardisation |
| Mg | Magnesium |
| MRL | Manufacturing Readiness Level |
| MRO | Maintenance, Repair, and Operations |
| MSG | Maintenance Steering Group |
| Muta. | Germ cell mutagenicity |
| NASA | National Aeronautics and Space Administration |
| NTSB | National Transportation Safety Board |
| Ni | Nickel |
| OEM | Original Equipment Manufacturer |
| OT&E | Operational Test and Evaluation |
| PU | Polyurethane |
| REACH | Registration, Evaluation, Authorisation and Restriction of Chemicals |
| R&D | Research and Development |
| SDS | Safety Data Sheet |
| SEA | Socio Economic Analysis |
| Skin. Sens. | Skin sensitisation |
| Skin irrit. | Skin irritation |
| STOT RE | Specific target organ toxicity, repeated exposure |
| SVHC | Substance of Very High Concern |
| TRL | Technology Readiness Level |
| UK | United Kingdom |
| µm | Micron |
| US | United States |

## Glossary

| Term | Definition |
|---|---|
| Active corrosion inhibition | The ability of a material to spontaneously repair small amounts of chemical or mechanical damage that exposes areas of metal without any surface protection (“self-healing properties”). |
| Adhesion promotion | Enhancement of the tendency of particles or surfaces to cling to one another (for example adhesion of coating to substrate, adhesion of paint to coating and/or substrate). |
| Aeroderivative | Parts used in power generation turbines used to generate electricity or propulsion in civil and defence marine and industrial applications that are adapted from the design/manufacturing processes and supply chains that produce parts for the aerospace industry. Typical applications include utility and power plants, mobile power units, oil and gas platforms and pipelines, floating production vessels, and for powering marine/offshore vessels such as Naval warships. |
| Aerospace | Comprises the civil aviation, defence/security and space industries. |
| Aerospace Companies | Companies principally engaged in carrying out the design, development, manufacture, maintenance, modification, overhaul, repair, or support of aerospace equipment, systems or structures, plus any aeroderivative uses. |
| Aerospace application | A single component in a single system for a single OEM’s hardware. Within a single OEM, even ostensibly ‘similar’ components or hardware in different systems / aircraft / engine models have unique design parameters. |
| Alloy | An alloy is a mixture of metals or a mixture of a metal and another element. |
| Alternative | A candidate alternative that has been tested, qualified, fully industrialised and certified by the Aerospace OEM. The definition is used only for the final classification of evaluated alternatives. |
| Certification | Verification that an aircraft and every part of it complies with all applicable airworthiness regulations and associated Certification Specifications (specs). |
| Chemical resistance | The ability of solid materials to resist damage by chemical exposure. |
| Coating | A covering that is applied to the surface of an object, usually referred to as the substrate. The purpose of applying the coating may be decorative, functional, or both. A coating may be organic (e.g. primer, topcoat or specialty coating) or inorganic (e.g. hard chrome, cadmium or zinc-nickel plating, thermal spray, anodize). |
| Conversion Coating | Chemical process applied to a substrate producing a superficial layer containing a compound of the substrate metal and an anion of an environment. Note that within the surface finishing industry a conversion coating is sometimes referred to as a passive coating or passivation. |
| Compatibility (with substrate/or other coatings) | The capability of two or more things to exist or perform together in combination without problems or conflict. In this document context usually refers to substrate, coatings or other materials and fluids |
| Corrosion | The process of an unwanted chemical reaction between a material and the environment, for example, oxidation of a metal part leading to loss of material. |

| Term | Definition |
|---|---|
| Corrosion inhibitors | Substances that retard corrosion by actively inhibiting corrosion reactions. |
| Corrosion protection | The methods employed in a system, which are used to minimize corrosion. |
| Corrosion resistance | The resistance a material offers against reaction with adverse environmental factors that can degrade the material. |
| Counterpart | Structural zone (like assembly, component) to which a given assembly/part is fitted. |
| Design parameters | The physical and environmental conditions and functional constraints derived from operational and customer requirements that are relevant for an aerospace component while in-service. Design parameters are specific to each individual component and each individual use of a component and vary between company and application. Design parameters include, but are not limited to, the hardware's base alloys; mating surfaces, including alloys, coatings, lubricants; exposure temperatures; structural stress and strain environment; fluid exposures; external environment including humidity, wind/rain erosion, etc.; functional characteristics; service life; etc. |
| Epoxy primer (stoved) | Base component of this type of primer is a thermally cross linked resin system which is responsible for key functionalities such as adhesion, chemical & thermal resistance, etc. |
| Implementation | After having passed qualification and certification, the third step is to implement or industrialise the qualified material or process in all relevant activities and operations of production, maintenance and the supply chain. |
| Legacy Part | Any part that is already designed, validated and certified by Airworthiness Authorities **or** for defence and space, any part with an approved design in accordance with a defence or space development contract. |
| Material Specification | Type of technical standard which has to be fulfilled by coating material. |
| Niche application | An aerospace application whose design parameters are different from the majority of other applications, such that the suitability for a given alternative will differ from the majority. |
| Potential Alternative | A possible alternative being evaluated for example in the labs of formulators. |
| Pre-treatment | Processes used to remove contaminates (e.g. oil, grease, dust), oxides, scale, and previously applied coatings (e.g. electroplated coatings, anodize coatings, conversion coatings, paint). The pre-treatment process must also provide chemically active surfaces for the subsequent treatment. |
| Qualification | OEM validation and verification that all materials, components, equipment or processes have to meet or exceed the specific performance requirements, defined in the Certification Specifications, documented in technical standards or specifications. |
| Risk reduction | Classification and labelling information of substances and products reported during the consultation being used for alternatives / alternative processes are compared to the hazard profile of the used chromate. |

## DECLARATION

We, *Indestructible Paint Limited*, request that the information blanked out in the “public version” of the Analysis of Alternatives is not disclosed. We hereby declare that, to the best of our knowledge as of today 21/07/17 the information is not publicly available, and in accordance with the due measures of protection that we have implemented, a member of the public should not be able to obtain access to this information without our consent or that of the third party whose commercial interests are at stake.

Signature: Date: 21/07/17

Alan Norton: Operations Director Indestructible Paint Limited; 16 – 25 Pentos Drive, Sparkhill, Birmingham, B11 3TA, U.K.

# 1. SUMMARY

## Introduction

This Analysis of Alternatives (AoA) forms part of the Application for Authorisation (AfA) for the use of pentazinc chromate octahydroxide (zinc tetrahydroxide chromate) in stoved epoxy primer for corrosion protection of aircraft engines components for aerospace and aeroderivative applications. The product, Rockhard Chromate primer containing pentazinc chromate octahydroxide, is formulated by Indestructible Paint Limited (IDP Ltd.) and used by OEMs in production and Maintenance Repair and Overhaul (MRO) activities. The AoA is based on input from European aerospace companies and formulators, with specific information from Rolls-Royce plc and its affiliates and Indestructible Paint The formulation of the Rockhard Chromate primer takes place at IDP Ltd. in Birmingham. Approximately, 0.06 tonnes of pentazinc chromate octahydroxide are used in Rockhard Chromate primer within the scope of this AfA per year.

Cr(VI) containing substances have been widely used since the mid-20th century. The multi-functionality of pentazinc chromate octahydroxide provides critical properties to the surfaces treated with the respective process. The pentazinc chromate octahydroxide-based surface treatments covered in this AoA for the aerospace industry are based on a stoved epoxy primer for the protection of Al alloys, Mg alloys and steel alloys in aircraft engine components, including related aeroderivative parts, which are used in civil and defence marine and industrial applications.

The treated components (e.g. fan cases, oil tanks, gearboxes, etc.) are safety critical parts of aircraft engines (see Figure 1) and therefore require an absolutely reliable and established treatment process. The most important key functionality of the Rockhard Chromate primer is to provide excellent corrosion protection which is highly dependent on the physical-chemical properties of the chromate compound, pentazinc chromate octahydroxide, explained in sufficient detail in chapter 3.3.1.

[redacted]

**Figure 1: Aircraft engine breakdown (Rolls Royce, 2017)**

Cr(VI), the active component in pentazinc chromate octahydroxide, has two-fold corrosion inhibition properties highly dependent on the substance's solubility. Firstly, it combines with the naturally occurring metal oxide to form a mixed valance chromium oxide layer that, by sitting on top of the metal, prevents oxygen from contacting the metal and thus provides a corrosion inhibition layer. Secondly, should the chromium oxide layer be damaged (e.g. by solid particle impact) then, after the initial creation of a thin metal oxide layer, the Cr(VI) ion, in its hydrated form, diffuses to the damaged region and re-establishes a corrosion inhibiting layer, albeit a layer that is less effective than before. To date, this "self-healing" mechanism (see Figure 2) appears to be unique to Cr(VI), and therefore not easily replaced.

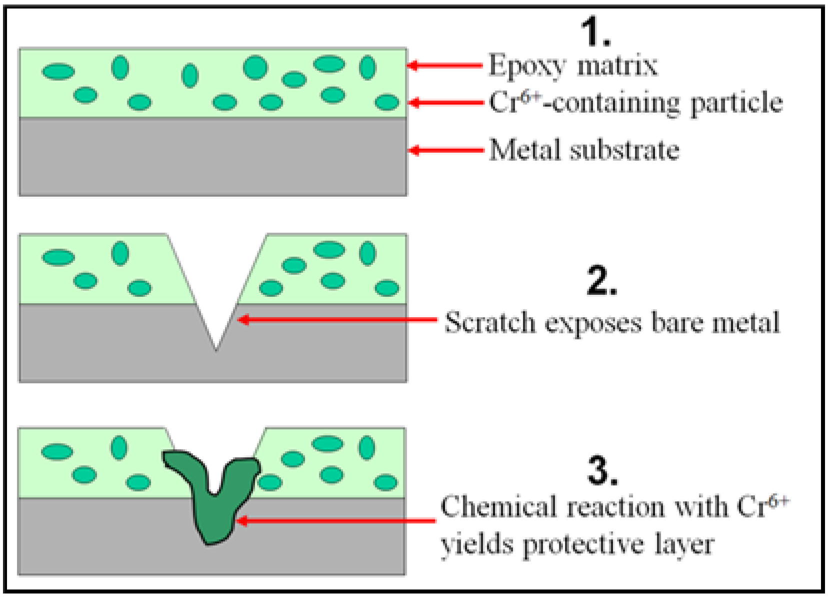


**Figure 2: Active corrosion inhibition (General Electric, 2013)**

As a result, chromate compounds in general have been considered the backbone of the corrosion protection scheme of aircraft engine components in aerospace and aeroderivative applications for several decades. The performance level of today's protection scheme is defined by this extensive history of the Cr(VI)-based coating systems currently being used.

**Key functionalities of the Rockhard Chromate primer**

The following key functionalities, sufficiently described in chapter 3.3.3, are crucial for aircraft engine components in aerospace and aeroderivative applications:

- Corrosion resistance: excellent corrosion protection and prevention to nearly all metals in a wide range of environments;
- Active corrosion inhibition: when a coating is damaged, e.g. by a scratch exposing the base material to the environment, the solubility properties of pentazinc chromate octahydroxide allow diffusion to the exposed area and inhibit corrosion;
- Adhesion promotion (i.e. adhesion to substrate and/or subsequent coatings or paint);
- Layer thickness;
- Chemical resistance (i.e. chemical resistance to hydraulic fluids)
- Temperature resistance (thermal shock resistance);
- Compatibility with substrates / other coatings;
- Flexibility (Bend); and
- Scratch resistance.

Various alternatives have been tested to substitute pentazinc chromate octahydroxide in Rockhard Chromate primer but none of them was found to be suitable to meet all requirements for this product, for each use, and specific design parameters, while also being technically and economically feasible.

**Use of the Rockhard Chromate primer containing pentazinc chromate octahydroxide**

Rockhard Chromate primer containing pentazinc chromate octahydroxide is specified in the aerospace industry because it provides superior corrosion resistance and inhibition (see chapter 3.3). These characteristics and the quality of the product are essential to the safe operation and reliability (airworthiness) of aerospace engine components, which operate under extreme environmental conditions sufficiently described in chapter 3.1.

The complexity of aircraft engines and the range of environmental conditions that these systems must withstand makes corrosion prevention a very challenging task. In practice, multiple coatings, such as pre-treatments, primers and top coats are specified to achieve the strict performance requirements (described above) necessary for regulatory compliance and for public safety in this industry, as described further below and in Annex A. Each coating system is different because it must meet individual functionalities and performance standards (i.e., design parameters) particular to a specific design. Figure 3 exemplarily depicts a typical structure of a Cr(VI)-based multi-layer coating system applied on aircraft engine components.

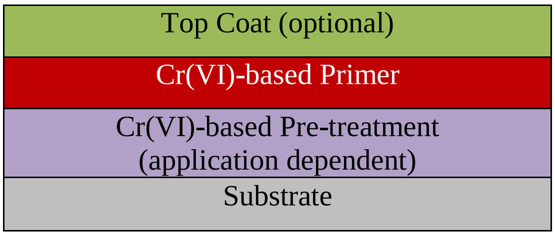


**Figure 3: Typical structure of a Cr(VI)-based multi-layer coating system applied on aircraft engine components.**

In general, the Rockhard Chromate primer containing pentazinc chromate octahydroxide is specified as one element of a complex system with integrated, often critical performance criteria. Compatibility with and technical performance of the overall coating system are primary considerations of fundamental importance.

Decades of service has built confidence in chromates performance for corrosion protection and other parameters across many differing technologies. This knowledge facilitates and enables exact lifetime predictions for component parts and therefore has significant influence on the level of risk created though substitution with alternative chemistries in safety-critical aerospace engines. Therefore, to minimise risk, a rigorous approach to research and design of alternative technologies is needed. A typical scheme for the development of Cr(VI)-free multi-layer coatings (e.g. primer or pre-treatment) is illustrated in Figure 4.

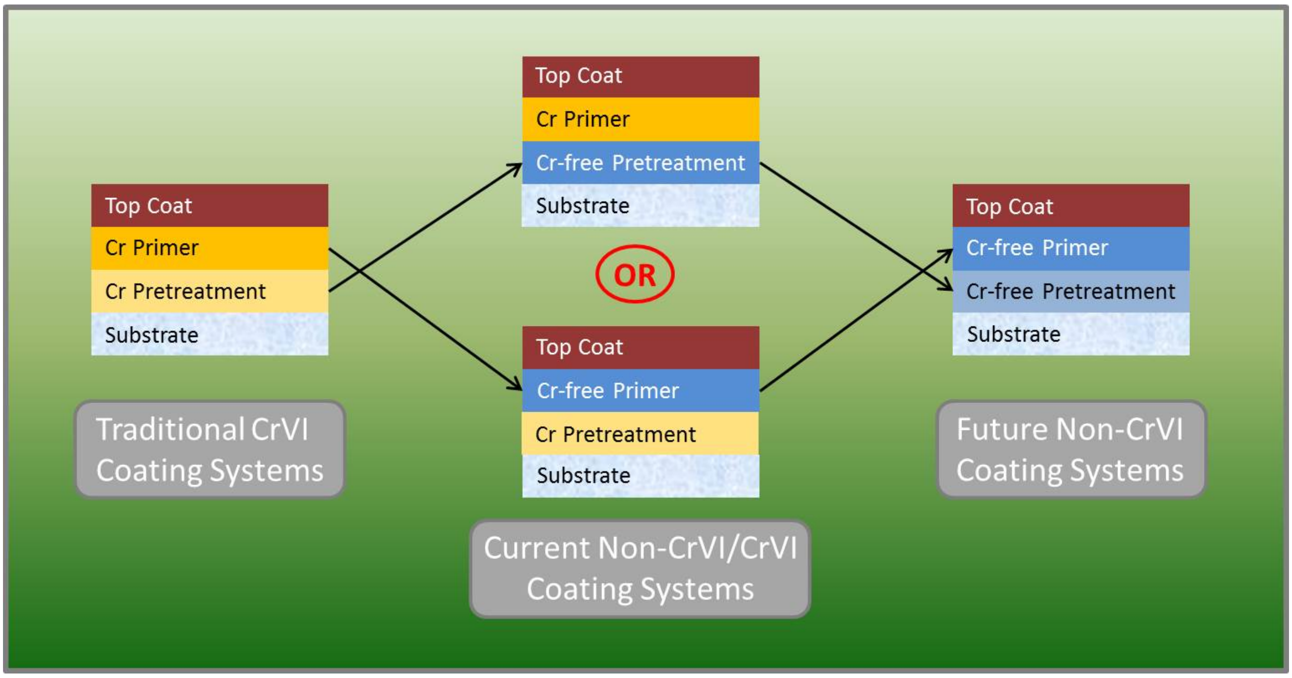


**Figure 4: Development of corrosion-prevention coating systems, from past to future (AMMTIAC, 2012, amended)**

Chromate coatings have been successively refined and improved as a result of over 50 years of research and experience in the aerospace industry, and reliable data are available to support their performance. While corrosion cannot be totally prevented, despite the highly advanced nature of Cr(VI)-based coating systems in place today, there is also extensive experience, amassed over decades, on the appearance and impact of corrosion to support its effective management in these systems.

Table 1 presents an overview of the three most recent and most promising alternatives to pentazinc chromate octahydroxide in Rockhard Chromate primer intensively investigated by IDP Ltd. However, at the current stage, it is not clear which candidate alternative(s), if any, will be successful and possibly implemented for aerospace applications within the scope of the AfA; and, at what point in time this may be the case. With flight safety at stake, the technical performance foremost must demonstrate “equal or better” capabilities with respect to critical design parameters.

**<u>Identification and evaluation of potential alternatives</u>**

In this AoA three alternative primer formulations for the substitution of pentazinc chromate octahydroxide are described in detail (see chapter 5.2.) and a full technical assessment was conducted by IDP Ltd. As of today, the candidates are tested for TRL4. In Table 1 an overview including technical assessment criteria of these alternatives is presented.

**Table 1: Overview of most promising alternative formulations for substitution of pentazinc chromate octahydroxide**

| Alternative primer formulation | Corrosion inhibiting substance | Technical limitation |
|---|---|---|
| LR2992 | [redacted] | Insufficient corrosion protection on Mg alloys |
| LR2965 | [redacted] | Insufficient corrosion protection on Al alloys |
| LR2964 | [redacted] | Insufficient corrosion protection on Al alloys |

The assessment of these alternative formulations shows that currently no technical equivalent to the state-of-the-art corrosion inhibitor pentazinc chromate octahydroxide is available. Therefore, **at least 14 years** of further R&D are necessary to develop and implement a suitable substitute for this substance. For a detailed description of the derivation of the length of the Review Period please refer to chapter 5.3.2.

**Ongoing development of potential Cr(VI)-free alternatives for aerospace applications**

Assuming a Cr(VI)-free alternative substance or mixture is identified as a result of ongoing R&D, extensive additional effort is needed beyond that point before it can be considered an alternative to pentazinc chromate octahydroxide in Rockhard Chromate primer used within the aerospace industry for application on safety critical engine components.

Aircrafts are one of the safest and most secure means of transportation, despite having to perform in extreme environments for extended timeframes. This is due to high regulatory standards and safety requirements set out by industry in close cooperation with authorities. The implications for substance substitution in the aerospace industry are described in detail in a report[1] prepared by ECHA and European Aerospace Safety Agency (EASA) in 2014, highlighting important arguments for long review periods for the aerospace sector based on the airworthiness requirements deriving from EU Regulation No 216/2008. Performance specifications defined under this regulation drives the choice of substances to be used either directly in the aircraft or during manufacturing and maintenance activities. It requires that all components, equipment, materials and processes incorporated in an aircraft must be certified, qualified and industrialised before production can commence. This process is illustrated in Figure 5. This system robustly ensures new technology and manufacturing processes can be considered 'mission ready' through a series of well-defined steps only completed with the actual application of the technology in its final form (and under mission conditions). When a

[1] http://echa.europa.eu/documents/10162/13552/aviation_authorisation_final_en.pdf

substance used in a material, process, component, or equipment needs to be changed, this extensive procedure must be followed in order to comply with airworthiness and other safety-driven requirements. The procedure for alternative development through qualification, validation, certification, industrialization and implementation within the aerospace industry is mirrored in the defence and space industries.

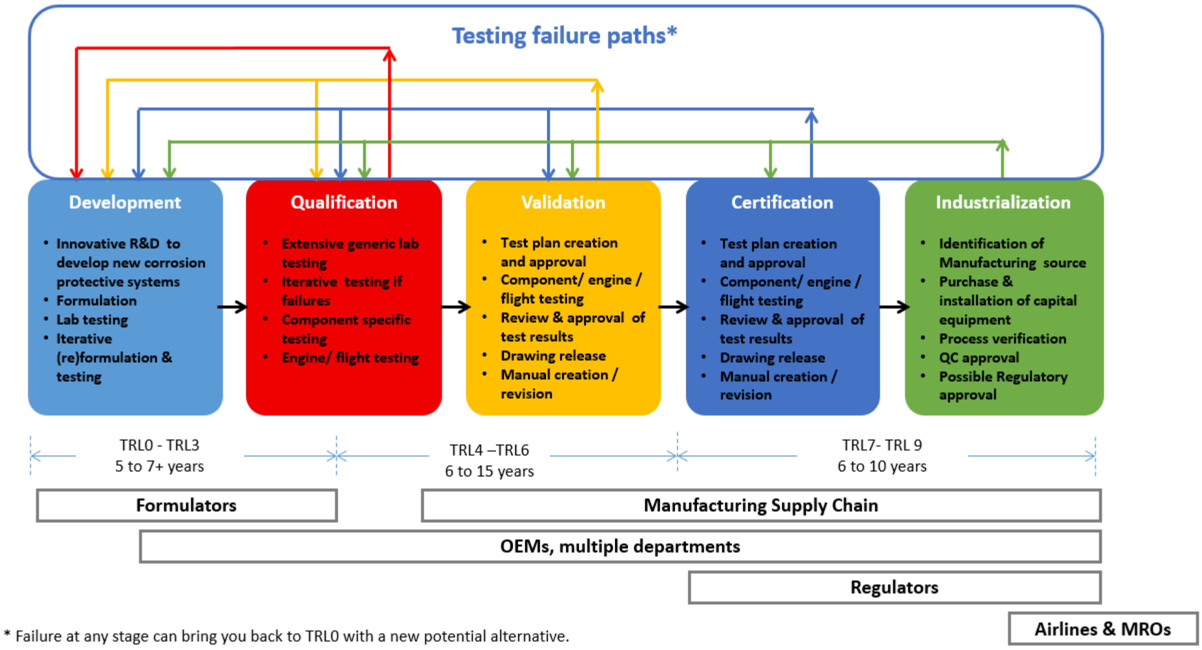


**Figure 5: Illustration of the development, qualification, validation, certification and industrialisation process required in the aerospace industry**

The detailed process involved in qualification, validation, certification, and industrialisation, and the associated timeframes, are elaborated in Annex A. Of course, these steps can only proceed once a candidate alternative is identified. A formulator does preliminary assessment of the viability of a potential alternative. Referring to experience, it can take 20 to 25 years to identify and develop a new alternative. While formulators are responsible for developing and performing the preliminary assessment of a potential alternative's viability, only the aerospace component / system design owner can determine when a candidate alternative is fully validated and certified for each of their aerospace applications. The testing criteria are determined on a case-by-case basis with due regard to the design and performance requirements of each component and system. Testing in a relevant environment over an appropriate timescale is necessary before a candidate alternative can be qualified and certified; this may require full engine and aircraft flight tests, even for very low volumes of product.

Validation and insertion of alternatives is even more challenging for existing hardware in-service or in-production than it is for new designs, as re-certification of all components incorporating the new processes and materials is required. Validation testing may be technically unjustified and economically unfeasible where the product is used in low volumes and under well-controlled conditions for aerospace applications.

Taken together, the alternatives assessed in this AoA show that at least 14 years are required to successfully substitute pentazinc chromate octahydroxide in Rockhard Chromate primer used on aircraft engine components for aerospace and aeroderivative applications. Although, there are promising approaches, none of them is mature enough to be used as a substitute and therefore additional time for R&D work is required. Additionally, the time intensive qualification, validation, certification, and industrialisation procedure for alternatives must also be taken into account when regarding the length of the review period.

As a further consideration, while the implications of the development process in the aerospace industry are extremely demanding, specification of an alternative, once available, can be built into the detailed specification for new aerospace systems. This is not the situation for existing systems, which may still be in production and/or operation. Production, maintenance and repair of these systems **must** use the processes and substances already specified following the extensive approval process. The Rockhard Chromate primer in the scope of this AfA is certified within the RR Material Specification MSRR9226. Substitution of pentazinc chromate octahydroxide in this specific product introduces yet another substantial challenge: re-certification of all relevant processes and materials produced with this specific specification.

In this context, the scale and intensity of IDP Ltd.'s R&D activities, conducted also in cooperation with other formulators, to identify alternatives to the pentazinc chromate octahydroxide primer is very relevant to the findings of the AoA. In general, serious efforts to find replacements for chromates have been ongoing within the aerospace industry for over 30 years and there have been several major programs to investigate alternatives to chromates in the aerospace industry over the last 20 years.

**Review Period**

The present AoA document shows the extensive R&D on potential alternatives to pentazinc chromate octahydroxide in Rockhard Chromate primer conducted by IDP Ltd. Furthermore, aspects of approval and industrialisation in the aerospace industry, are assessed with regard to a future substitution of the substance. The following key points are relevant for deriving the review period:

- Extensive R&D on viable alternatives to pentazinc chromate octahydroxide in Rockhard Chromate primer has been carried out over the last few decades but did not lead to the development of substitutes for the applications within the scope of this AfA that could be available within the normal review period. The unique functionalities of Cr(VI) compound make it challenging and complex to replace the substance in surface treatment, especially regarding applications where superior corrosion is crucial for public safety;
- Because of the lack of potential pentazinc chromate octahydroxide alternatives for aerospace and aeroderivative applications, a **review period of at least 12 years** is needed to identify, qualify, validate, certify, and industrialise alternatives. A short or normal review period will place a disproportionate and unjustified administrative and economic impact on the aerospace industry, with important consequences for the supply chain;
- The aerospace investment cycle is demonstrably very long. Aerospace components are usually designed to allow an overall lifetime of 20 to 30 years or more for civil and industrial uses and 40 to > 90 years for defence uses before they are finally taken out of service (see SEA). Therefore, it is technically and economically meaningful to substitute only when a major redesign or refurbishment takes place;
- Any alternative is required to pass full qualification, validation, certification and implementation / industrialisation to satisfy specific performance criteria to comply with regulations and requirements (e.g. airworthiness regulations) and to ensure safety of use (including the necessary certifications for aerospace components using the alternative). Before any potential alternative can be implemented, aerospace components are required to comply with all applicable regulations and associated Certification Specifications (e.g., EASA CS-25 and EASA CS-E);
- Comparing health impacts of workers to socio-economic impacts, the ratio in the baseline scenario is at least 1:658 (see chapters 7.1 and 7.2 of the SEA), and there is clear evidence that this situation is not likely to change in the next decade.

**Concluding remarks**

An extensive amount of research over the last 30 years has been deployed to identify and develop viable alternatives to Cr(VI)-based primers. Due to its unique functionalities and performance, it is challenging and complex to replace surface treatments based on pentazinc chromate octahydroxide in aerospace applications that demand superior performance for especially corrosion and also adhesion to deliver safety over extended time periods and extreme environmental conditions. Candidate alternatives are under intensive investigation by IDP Ltd. Based on efforts to date, it is anticipated that **a review period of at least 12 years** is needed to qualify, certify and industrialise a suitable alternative for the relevant applications in the aerospace industry.

# 2. INTRODUCTION

## 2.1. Substance

The following substance is subject to this AoA:

Table 2: Substance of this analysis of alternatives.

| Substance | Intrinsic property(ies)[1] | Latest application date[2] | Sunset date[3] |
|---|---|---|---|
| Pentazinc chromate octahydroxide<br>EC No: 256-418-0<br>CAS No: 49663-84-5 | Carcinogenic (category 1A) | 22.07.2017 | 22.01.2019 |

[1] Referred to in Article 57 of Regulation (EC) No. 1907/2006
[2] Date referred to in Article 58(1)(c)(ii) of Regulation (EC) No. 1907/2006
[3] Date referred to in Article 58(1)(c)(i) of Regulation (EC) No. 1907/2006

This substance is categorised as substance of very high concern (SVHC) and is listed on Annex XIV of Regulation (EC) No 1907/2006. It is an inorganic chromate salts based on hexavalent chromium (Cr(VI)). Adverse effects are discussed in the CSR.

The applicant Indestructible Paint Limited (IDP Ltd.) applies for authorisation to continue the use of pentazinc chromate octahydroxide in primer applications for protection of Al alloys, Mg alloys and steel alloys in aircraft engine components for aerospace and aeroderivative applications. More specifically, IDP Ltd. uses this substance only in the product Rockhard Chromate primer, which therefore can be regarded as synonym for the pentazinc chromate octahydroxide compound.

The aim of this analysis of alternatives is to demonstrate that no feasible alternatives to pentazinc chromate octahydroxide **will be available before 2031.**

## 2.2. Uses of pentazinc chromate octahydroxide

Cr(VI) containing substances have been widely used since the mid-20th century. The multi-functionality of pentazinc chromate octahydroxide provides critical properties to the surfaces treated with the respective process. The pentazinc chromate octahydroxide-based surface treatments covered in this AoA for the aerospace industry are based on a stoved epoxy primer for the protection of Al alloys, Mg alloys and steel alloys in aircraft engine components, including related aeroderivative parts, which are used in civil and defence marine and industrial applications.

Aeroderivative parts are adapted from the design / manufacturing processes and supply chains that produce parts for the aerospace industry. Aeroderivative products make up a small percentage of the total aircraft hardware volume in the European Union (EU) and are used to generate electricity or propulsion in civil and defence marine, oil and gas, and industrial applications.

The Rockhard Chromate primer is applied on different component parts of aircraft engines listed in Table 3. The use of pentazinc chromate octahydroxide in Rockhard Chromate primer is mandatory for these applications (e.g. as per Rolls Royce Material Specifications).

Table 3: Scope of the AoA

| **Product** | Stoved epoxy primer |
|---|---|
| | Production and MRO |
| **Part family** | Aircraft engine components for aerospace and aeroderivative applications |
| **Substrate** | Aluminium alloys, Magnesium alloys and steel alloys |
| **Parts** | 98 component examples are listed below:<br>- Compressor casings,<br>- Fan cases<br>- Oil tanks (internal and external)<br>- Gearboxes,<br>- Couplings,<br>- Transmissions,<br>- Oil filter housings,<br>- Fuel filter covers,<br>- Plugs,<br>- Washers,<br>- Plates<br>- Front Frame<br>- MRO applications |

Please note that this list is not exhaustive. The part designs will be common across the variants within the same product and other engine products.

### 2.3. Purpose and benefits of Cr(VI) compounds

Pentazinc chromate octahydroxide offers a broad range of functions, mainly based on the characteristics of the Cr(VI) compound. It has been widely used for over 50 years in the aerospace industry in various applications. The multi-functionality of pentazinc chromate octahydroxide provides critical properties to the surfaces treated with the respective process. The following key functionalities, sufficiently described in chapter 3.3.3, are crucial for aircraft engine components in aerospace and aeroderivative applications:

- Corrosion resistance: excellent corrosion protection and prevention to nearly all metals in a wide range of environments;
- Active corrosion inhibition: when a coating is damaged, e.g. by a scratch exposing the base material to the environment, the solubility properties of pentazinc chromate octahydroxide allow diffusion to the exposed area and inhibit corrosion;
- Adhesion promotion (i.e. adhesion to substrate and/or subsequent coatings or paint);
- Layer thickness;
- Chemical resistance (i.e. chemical resistance to hydraulic fluids)

- Temperature resistance (thermal shock resistance);
- Compatibility with substrates / other coatings;
- Flexibility (Bend); and
- Scratch resistance.

Various alternatives have been tested to substitute pentazinc chromate octahydroxide. However, it is a challenging task to find a substitute which meets all requirements for a product, for each use, and specific design parameters, while also being technically and economically feasible.

### 2.4. MRO business in the aerospace sector

MRO is a central concept in the aerospace sector. Aircrafts are usually dimensioned to allow an overall lifetime of 20 to 30 years before they are finally taken out of service. An aircraft engine is exposed to extreme mechanical forces and temperatures as well as to conditions that give rise to hot gas corrosion caused by sulphur and oxidation. Therefore the building components of aircraft engines are subjected to corrosion, wear, oxidation, vibrations and fatigue, as the required performance of the parts inevitably suffers and, if no adequate measures are taken, can be subject to failures. Moreover there are so called "life limited parts" that are deliberately operated above their yield strength, for example compressor and turbine discs and blades. Thus, in order to maintain flight security, aircraft are subject to intensive MRO activities.

Importantly, the disposal of “used” components and exchange for new parts is not common practice in the aerospace sector, as most of the parts are just too big and too expensive.

The MRO activities underlie national and international requirements that cover the different types of aircraft as well as specific components. The maintenance programme is strictly scheduled in the manufacturer’s maintenance manual that has to be officially approved by the authorities. The method for repair of the parts is also specified by the OEMs in the maintenance manual in the “service bulletins”.

**Please note that MRO facilities can only change the materials used in MRO activities under the strict approval of the approved design organisation (the OEM that holds the Type Certificate), in accordance with Commission Regulation (EU) No 1321/2014. Before such an approval can be granted, the design organisation must first assure itself that the alternative (e.g. primer formulation) is acceptable as an alternative without detriment to material compatibility, service life, product safety, reliability, etc. In practice, it will be impractical and uneconomical to introduce such changes for many such aerospace systems.**

Introducing a new part to an established system or assembly is complicated, as the interactions of complementary parts are very complex. Even a presumed improvement in one component is not necessarily beneficial for the whole system or assembly. Changing one detail of a part in a system or assembly can lead to unanticipated failures. The more complex a system, the more likely it is that small changes can have significant unforeseen impact. Evaluation of the complex interplay of the complementary parts needs to be re-evaluated, entailing long approval and industrialisation periods, which can take years to implement.

It is the aim of the MRO activities to restore the original function of the equipment and components. In this regard, the usage of Rockhard Chromate primer containing pentazinc chromate octahydroxide is also used at MRO facilities to restore the safety relevant corrosion protection system of individual engine components that have been distorted by due to the demanding environments (e.g. temperature, humidity, stress, etc.) they are exposed to. Importantly, the repair treatments must be conducted in accordance with the OEM’s specifications, as aircraft engines are highly complex systems where even small changes may have significant negative impacts on the engine. In Annex A, more information is provided on the extensive development and approval procedures that are necessary to implement any changes in the aerospace sector.

# 3. ANALYSIS OF SUBSTANCE FUNCTION

## 3.1. Functional overview

The predominant uses of the pentazinc chromate octahydroxide-based primer are to provide corrosion resistance, improved adhesion, and improved chemical resistance. Inspection and maintenance of hardware to assure integrity is a challenging task due to location and complexity. Various levels of maintenance are continuously required based on the inspection periods and the hardware use.

Metal surfaces on aircraft engine components face a broad variety of exposures, which result in corrosion:

- temperature
- humidity
- salt
- erosion (volcanic ash, due to debris, damage, etc.)
- radiation
- stress
- pressure
- accumulated liquids (water, spilled food, human waste, acid rain, etc.)
- operational fluids (fuel, hydraulic oil, de-icing, etc.)
- galvanic coupling (dissimilar metal contact)

All these factors can occur alone or in combination in aircraft engine components, increasing the corrosion risk. The Rockhard Chromate primer, containing pentazinc chromate octahydroxide, in the scope of this AfA, is applied on engine component parts mentioned in Table 4. Please note that this list is not exhaustive.

**Table 4: Engine component parts treated with Rockhard Chromate primer**

| **Part family** | Aircraft engine components for aerospace and aeroderivative applications |
|---|---|
| **Parts** | Compressor casings<br>Fan cases<br>Oil tanks (internal and external)<br>Gearboxes<br>Couplings<br>Transmissions<br>Oil filter housings<br>Fuel filter covers<br>Plugs<br>Washers<br>Plates<br>Front Frame |

| | MRO applications |
|---|---|

Importantly, in this demanding environment corrosion still occurs with the highly developed Cr(VI)-containing coating systems. Decades of extensive in-service experience under real operating conditions relating to the appearance and impacts of corrosion on Cr(VI) systems allows the aerospace industry to define inspection, maintenance, and repair intervals.

Without a well-developed Cr(VI)-free alternative, corrosion will increase, as these alternative coatings do not offer all the crucial properties of Cr(VI)-based coating systems. Their long-term performance can currently only be estimated, resulting in significantly shorter inspection, maintenance, and repair intervals.

Corrosion issues likely would not appear suddenly, but after several years when thousands of aerospace components are in-service. Further, potential decreased corrosion protection performance from Cr(VI)-free coatings would necessitate shorter inspection intervals to prevent failures. Flight safety obligations preclude the aerospace industry from introducing inferior alternatives.

In the purely hypothetical case where decreased or loss of corrosion protection is introduced to aircraft, the following risk mitigation actions may be required:

- Substantial increase in inspections – both visual checks, and non-destructive evaluations, such as ultra-sound crack tests, etc. Some inspections are very difficult or hazardous to perform (e.g. inside fuel tanks or inside fuselage / wing structures). All aircrafts using less effective materials in repair / overhaul and/or unproven materials across the operating lifetime of an aircraft would be subject. A very conservative inspection frequency (i.e. short time or flight cycle interval) would be set to ensure safety until adequate in-service performance experience is obtained.
- Increased overhaul frequency or replacement of life-limited components.
- Possible early retirement of aircraft due to compromised integrity of non-replaceable structural parts.
- Whole fleets may be grounded until a repair / replacement plan is in place for the whole aircraft fleet
- Due to similarity of technologies and aircraft uses, a fleet grounding event in such a scenario could impact many or all aircraft fleets.
- An increase in the number of aircraft required by each airline would be needed to compensate for inspection / overhaul downtime and early retirement.

- Defence systems would have similar impacts adversely affecting operational availability, with consequences for national security and equipment procurement.

Aircraft engine components are parts of major systems and the hardware in these systems are designed to last the common service interval between major overhauls. For example, a system is designed to achieve 25,000 cycles between overhauls and a new component is rated for 15,000 cycles because of a Cr(VI)-free coating. By default, the entire system would now be overhauled every 15,000 cycles. Take a casing as an example. If that casing can only survive for a portion of the life of an engine due to limitations of a new coating, the engine would require disassembly. This means taking the engine off the wing, sending it to a repair centre, disassembling the engine and replacing the parts at much shorter intervals than needed for the remainder of the engine; thus adding inherent inspection, maintenance, and repair costs to the manufacturers, operators, and end use customers. The shortening of overhaul intervals therefore has a substantive impact on the costs of existing and new aircraft engine maintenance contracts, impacting the overall cost of ownership of every aircraft engine affected.

The lack of experience with Cr(VI)-free solutions can have a critical safety impact. The Aloha Airlines incident from 1988 demonstrates the significant consequences of corrosion and the quality and surveillance of airline maintenance programmes. In this case, corrosion ultimately led to loss of a major portion of the upper fuselage in full flight at 24,000 feet, near the front of the plane. Multiple corrosion-initiated fatigue cracks were detected in the remaining aircraft structure located in the holes of the upper row of rivets in several fuselage skin lap joints. Safety issues raised in the official accident report (NTSB/AAR-89/03) included: “the quality of air carrier maintenance programs and the FAA surveillance of those programs, the engineering airworthiness of the B-737 with particular emphasis on multiple site fatigue cracking of the fuselage lap joints, the human factors aspects of air carrier maintenance and inspection for the continuing airworthiness of transport category airplanes, to include repair procedures and the training, certification and qualification of mechanics and inspectors.”

While this incident did not involve a Cr(VI)-free coating, it provides valuable insight into potential consequences if a Cr(VI)-free protection system were to be introduced without establishing adequate inspection, maintenance, and repair intervals. Despite the best knowledge today, hidden properties or incorrect performance predictions of Cr(VI)-free systems cannot be excluded and remaining risks must be mitigated. Ultimately, extensive qualification and validation testing is not equivalent to 50 years real-life experience with Cr(VI)-based corrosion protection.

The following links provide other examples of corrosion-related failures across industries (aerospace, shipping, civil engineering, and oil and gas).

- National Transportation Safety Board (NTSB) - https://www.ntsb.gov/_layouts/ntsb.aviation/brief.aspx?ev_id=20100520X02527&key=1
- UK Air Accidents Investigation Branch - https://www.gov.uk/government/organisations/air-accidents-investigation-branch
- Corrosion Doctors - http://corrosion-doctors.org/Forms/Accidents.htm

The aerospace industry has a permanent learning loop of significant events and failure analysis and decisions for safety improvements. Part of this improvement is the introduction of the Maintenance Steering Group 3 Analyse (MSG-3), specifically developed for corrosion. MSG-3 provides a system for OEMs and the regulators to identify the frequency of inspection with respect to the stress corrosion, protection, and environmental ratings for any component or system. Without adequate experience, proven success, and therefore possible unknown or hidden properties, the performance of a Cr(VI)-free system cannot be highly rated. Consequentially, a significant reduction to the maintenance interval is required, potentially cut in half. For cases with no long-term experiences or correlation to in-service behaviour, which is normally the case, a further reduction to the maintenance interval may be required.

As a current example, in 2010 the costs for an aircraft heavy structural inspection were $425K to $4.5M based on the aircraft model. Performance of these checks is normally every four to 12 years. If the inspections increase to every two to six years, the financial dimension of such a change becomes evident. Therefore, to determine proper inspection intervals, a detailed knowledge of the alternative's performance is a prerequisite. Some examples on parts where pentazinc chromate octahydroxide is applied are illustrated in Figure 6.

[redacted]

**Figure 6: Example for parts (fan cases) on which the Rockhard Chromate primer is applied (Rolls Royce, 2017)**

## 3.2. Surface treatment process

Surface treatment of metals is a complex, systematic process. For operations with high performance requirements in demanding environments, the use of Cr(VI)-containing treatments is essential to ensure the decades-long quality and safety of the end product.

However, only the combination of adequate pre-treatment, main process and post-treatment leads to a well-prepared surface providing all necessary requirements for the respective design parameters, as described in chapter 3.3.2 and 3.3.3. The usage of Rockhard Chromate primer, containing pentzinc chromate octahydroxide, in the main treatment process is crucial to ensure the quality of the aircraft engine components and meet the safety and performance requirements of the aerospace industry.

The importance of surface preparation cannot be overemphasised in obtaining an excellent protective primer coating. The role of a primer in coating protection is to isolate the substrate from the environment. Therefore, the surface of the substrate on which the coating system should be applied to primer is being applied should be critically clean, in order to achieve the necessary adhesion.

In general, primers act as permeable membranes through which oxygen and moisture can diffuse and so the inhibition of an oxidation reaction is due to their chemical inhibition in which the chromate pigments act as anodic passivates and stabilise the oxide film.

The selection of a specific pre-treatment and/or primer system will always dependent on the substrate material, required application and the operating environment.

### 3.2.1. Primers

Primers are a pigmented composition of liquid consistency applied as a thin layer which converts to a solid, adherent and tough film.

Rockhard Chromate primer with pentazinc chromate octahydroxide is used for metallic materials such as Al-, Mg- and steel alloys. In context of this dossier, the primer is a corrosion-inhibiting coating which is applied on various substrates or treated surfaces.

In general, primers are low viscosity dispersions of solid components in a blend of various liquids and are based on the following main components:

- The first component of the kit is the binder or base. It serves as the matrix, and is usually composed of a synthetic resin. Typical resins are epoxies, alkyds and PUs due to their excellent adhesion properties and resistance to exposure to a range of aviation fluids. The pentazinc chromate octahydroxide is dispersed and held in suspension in the liquid binder.
- The second component of the kit is the catalyst. In multicomponent products, the resin will be cured/crosslinked by combining it with a curing agent such as a polyisocyanate or polyamine. The rate of the reaction is usually controlled by the catalyst.
- The third component is the solvent or thinner. This can be either an organic solvent, water, or a combination of both. The thinner controls the viscosity of the liquid/solid dispersion. Solvents represent a volatile component and evaporate into the atmosphere after the primer is applied.

Besides the three major components mentioned above other additives may be used as part of the formulation to control, for example, the rheology of the dispersion (i.e. the liquid flow over the component surface after primer application), the rate of reaction in a multicomponent system, the adhesion to a range of substrates or the flow and surface wetting of the applied product.

Aircraft engine components consist of a multilayer system that is illustrated below (Figure 7). This surface structure is crucial to deal with the complexity of an aircraft and the airworthiness requirements that make corrosion resistance and improved paint adhesion a very challenging task. The primer constitutes one layer out of several layers of coating applied to the surface of an aeronautic vehicle or component. The multi-layer system were the Rockhard Chromate primer is applied is specifically described in chapter 3.3. A typical and simplified Cr(VI)-based multi-layer coating system is provided in Figure 7.

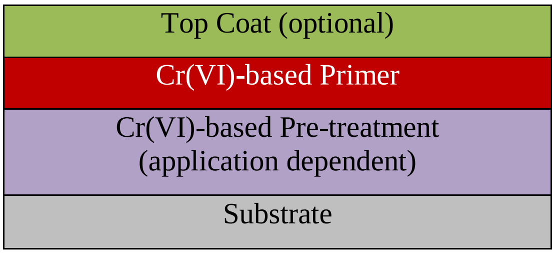


**Figure 7: Typical structure of a Cr(VI)-based multi-layer coating system applied on aircraft engine components.**

For several decades chromates have been the backbone of corrosion protection in the aerospace sector. The performance level of today´s corrosion protection scheme is a result of and continues to be defined by this extensive history and development of the Cr(VI)-based coating systems currently being used.

Furthermore, it is critical to appreciate that a particular corrosion protection scheme applicable for one component in one aircraft cannot be applied in any other component in the same aircraft or any other aircraft unless it has been qualified according to airworthiness requirements.

**This means that even if an alternative has been approved in one component in one aircraft-model, it cannot be inserted into what appears to be the same part in another aircraft until it is fully reviewed/validated/certified to ensure the performance of the alternative is fully acceptable under the unique operating and environmental conditions for each 'design application'. Furthermore, extensive experience shows that an alternative that is finally successfully certified in one component in one model cannot necessarily be successfully certified in another. Circumstances for each component in each model are unique and extrapolation is generally not possible.**

The primer supplied by IDP Ltd. and applied to various component parts of aircraft engines for aerospace and aeroderivative applications is described in more detail in the following chapter 3.3**.**

### 3.3. Rockhard Chromate Primer

The Rockhard Chromate primer with pentazinc chromate octahydroxide is a stoved epoxy primer which is formulated by IDP Ltd. and used for the application on aircraft engine component parts to primarily provide excellent corrosion resistance. Rockhard Chromate primer is used in production and MRO activities. It is approved (e.g. according to the Rolls Royce Material specifications) and therefore used across multiple substrates (Al-, Mg- and steel alloys) as part of different coating systems described below. In summary its multi-substrate and multi-system use has not been able to be replicated across all of its requirements by one single chrome-free product. Examples for typical coating systems on different substrates are provided below.

<u>Variant A</u>

| |
|---|
| Top Coat |
| Rockhard Chromate primer |
| Cr(VI)-based pre-treatment |
| Substrate |

Variant A is typically used as a corrosion protection system on magnesium based gearboxes.

<u>Variant B</u>

| |
|---|
| Rockhard Chromate primer |
| Cr(VI)-based pre-treatment |
| Substrate |

Variant B is for example used on the AE3003 front frame.

<u>Variant C</u>

| |
|---|
| Top Coat |
| Rockhard Chromate primer |
| Substrate |

Variant C is typically used on aluminium based transmission systems or sub-systems which come in contact with oil. An additional application example of this coating configuration are the external surfaces of oil tanks.

<u>Variant D</u>

| |
|---|
| Rockhard Chromate primer |
| Substrate |

Variant D is almost exclusively used on aluminium based fan cases where pre-treatment process are precluded. For example the Trent700 and RB211 fan cases made from corrosion prone 2000-series aluminium alloys that are alloyed with high levels of copper (approximately 4-6 %) are typically applications of this configuration. Due to the size and required mechanical integrity of the fan case, no pre-treatment to improve the poor corrosion-resistance of the native aluminium substance can be undertaken, therefore protection is solely reliant on the stoved epoxy primer. Further application examples are oil tanks (internal surfaces) and compressor cases.

The chemical composition of the Rockhard Chromate primer is described in Table 5 below.

**Table 5: Rockhard Chromate primer: chemical composition and functionality of components**

| Substance / Compound (CAS no.) | Percentage in Primer |
|---|---|
| Barium chromate (10294-40-3) | 10 - 20 % |
| Xylene (1330-20-7) | 10 - 20 % |
| Butyl acetate (123-86-4) | 10 - 20 % |
| 4-Hydroxy-4-methylpentan-2-one (123-42-2) | 5 - 10 % |
| Butyl glycol (111-76-2) | 5 - 10 % |
| Solvent Naphtha (petroleum), heavy arom.; kerosin - unspecified (64742-94-5) | 5 - 10 % |
| 1-Methoxy-2-propanol (107-98-2) | 3 - < 5 % |
| Melamine P/W formaldehyde butylated (68002-25-5) | 1 - < 3 % |
| 4-Methylpentan-2-ol (108-11-2) | 1 - < 3 % |
| Propan-1-ol | 1 - < 3 % |
| **Pentazinc chromate octahydroxide (49663-84-5)** | **< 1 %** |
| Pine oil (94266-48-5) | < 1 % |
| 2-Methoxy-1-methylethyl acetate (108-65-6) | < 0.2 % |
| Strontium chromate (7789-06-2) | < 0.1 % |
| Naphthalene (91-20-3) | < 0.1 % |
| Phthalic anhydride (85-44-9) | < 0.1 % |
| Formaldehyde (50-00-0) | < 0.1 % |

### 3.3.1. Pentazinc chromate octahydroxide - substance specific characteristics

The substance that is in the scope of this AoA, pentazinc chromate octahydroxide, is used as corrosion inhibitor in the formulation of the Rockhard Chromate primer product. Its corrosion inhibiting properties are extremely effective at low loadings (i.e. concentrations), as explained below. Corrosion inhibitive particles of pentazinc chromate octahydroxide are only effective in solution and therefore the level of solubility is highly related to the protection against corrosion.

In order to provide the desired functionality (see chapter 1.1.2 below), pentazinc chromate octahydroxide has to be available in sufficient quantity and mobility to reach an unprotected scratched area on the material in sufficient concentration to modify or prevent the corrosion process.

Importantly, Cr(VI) compounds cannot necessarily be used interchangeably, as they may have important variations in physical-chemical properties (such as pH, solubility, etc.) and other differences. For example, the substances that have limited solubility in water, such as pentazinc chromate octahydroxide, potassium hydroxyoctaoxodizincatedichromate and strontium chromate, are used in primers as corrosion inhibitors. In contrast to that, the more soluble substances, such as sodium or potassium dichromate, are used in metal finishing baths e.g. anodize seals. Furthermore, different Cr(VI) compounds are used for different primer applications as for example compared to potassium hydroxyoctaoxodizincatedichromate, the solubility of pentazinc chromate octahydroxide is considerably lower.

### 3.3.2. Key Chromate functionalities - active and passive corrosion protection

The basic functions of a chromate based surface protection system are the combination of the physical barrier function provided by the passive layers and the active features of chemical corrosion inhibition of the free surface after damage, which is illustrated in Figure 8 and described below:

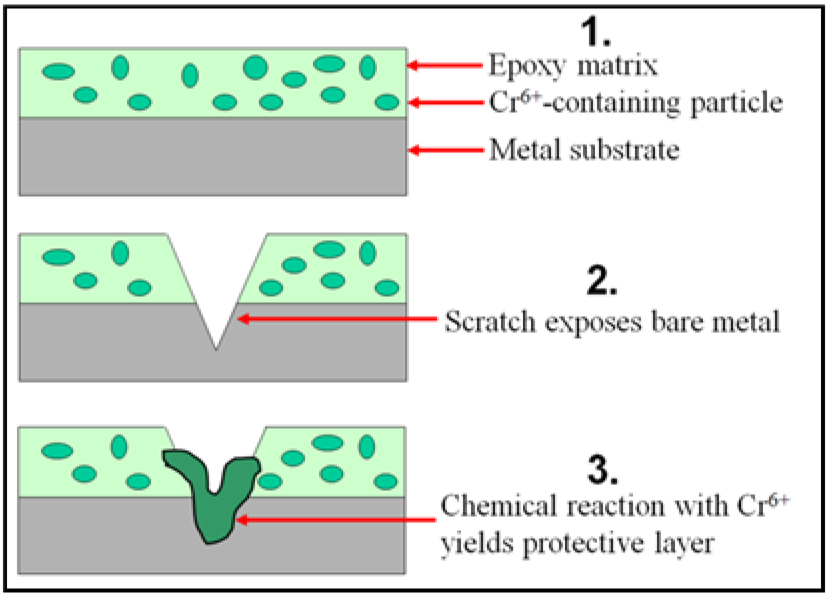


**Figure 8: Active corrosion inhibition (General Electric, 2013)**

Cr(VI) has two-fold corrosion inhibition properties. Firstly, it combines with the naturally occurring metal oxide to form a mixed valance chromium oxide layer that, by sitting on top of the metal, prevents oxygen from contacting the metal and thus provides a corrosion inhibition layer (the principle is not dissimilar to that of stainless steel where surface oxides, of Ni and Cr, prevent oxygen attacking the base metal, Fe). Secondly, should the chromium oxide layer be damaged then, after the initial creation of a thin metal oxide layer, the Cr(VI) ion, in its hydrated form, diffuses to the damaged region and re-establishes a corrosion inhibiting layer, albeit a layer that is less effective than before. The areas close to the damaged region will become depleted in Cr(VI), thus reducing the corrosion protection offered in the immediate area. However, the diffusion mechanism operates continuously, allowing further diffusion of Cr(VI) ions from more distant areas into the depleted area. This dynamic process represents the “self-healing” mechanism that to date appears to be unique to Cr(VI).

Chromates in general, are unique with respect to the efficiency / concentration ratio not achieved with other compounds. The Cr(VI) atom owes its unique ability to inhibit corrosion to its physical-chemical properties. No other element is as effective in these specific characteristics. These unique functionalities of Cr(VI) make it an ideal and not easily replaceable substance. Moreover, their solubility and release-rate can be adjusted by selection of the cation. As a result, chromates have been considered the backbone of the corrosion protection scheme of aircraft engine component parts in aerospace and aeroderivative applications for several decades. The performance level of today’s protection scheme is defined by this extensive history of the Cr(VI)-based coating systems currently being used.

Pentazinc chromate octahydroxide has been determined to be extremely effective in protecting discontinuities in coating films against corrosion. Performance tests and routine quality control tests show that the solubility of pentazinc chromate octahydroxide is sufficient to provide an adequate supply of corrosion inhibiting species (active and passive conservation).

The protection requirements for aerospace components are very demanding. The characteristics of pentazinc chromate octahydroxide have passed the specification thresholds to be applied in the Rockhard Chromate primer for the application in aerospace systems and have been proved to be effective against corrosion.

### 3.3.3. Key technical criteria

A multitude of aircraft engine components are in the scope of this AfA as their performance relies on the treatment with pentazinc chromate octahydroxide. A major challenge in preparing this AoA is therefore to identify and summarize key functionalities and corresponding requirements across this multitude of parts and to present a representative feasibility assessment of potential alternatives. Performance requirements for current surface treatments, exemplarily described in chapter 3.3, are set out by RR in relevant Material Specifications, which contain the individual Rockhard Chromate primer application on the different engine component parts.

- In this case, the most important specification requirement is corrosion resistance;
- When a new alternative is tested, not all functionalities are tested in parallel from the beginning. R&D will first focus on the most important functionalities, which are described in

Table 6 below. Typically, first tests to be initiated are for corrosion resistance, as the most important functionality for aerospace applications besides adhesion;

- Testing on other key functionalities is initiated more or less in parallel to the long lasting corrosion testing. This is done to speed up the development process and to not lose valuable R&D time if the alternative meets the requirements for corrosion resistance. However, if corrosion protection fails in early stage, no further testing is conducted on other functionalities.

Please note that Table 6 is not exhaustive but serves to highlight the key functionalities for the applications that are in the scope of this AfA. Depending on the product (e.g. gear box component part) and its specific application, further important functionalities besides those listed in Table 6 may need to be taken into account. The key technical criteria which affect the suitability of alternatives to using pentazinc chromate octahydroxide in Rockhard Chromate primer are listed in Table 6.

**Please note that a potential alternative satisfying the qualification criteria described in Table 6 does <u>not</u> make the alternative a certified substitute to Rockhard Chromate primer containing pentazinc chromate octahydroxide until at least equivalency with no impact on service life under actual use conditions is proven.**

**Table 6: Quantifiable key functionalities and requirements of Rockhard Chromate primer**

| Key functionality | Requirements | Test Method and Criteria |
|---|---|---|
| Corrosion resistance | -3000 hours active corrosion protection for Al alloys in hot and cold environments<br>-500 hours active corrosion protection for Mg alloys in hot and cold environments | Neutral salt spray test: **ASTM B 117** or **BS EN ISO 9227.**<br>Cyclic heat / salt spray: 10 cycles of the following cycle: 2 hr at 220 °C, 20 hr in salt spray to **ASTM B 117** or **BS EN ISO 9227**.<br>→Coating must not become detached or damaged under test conditions |
| Adhesion | High bond strength when applied in direct to metal. | **BS EN ISO 2409**: Classification 2 or below.<br>**ASTM D 3359** (Method B): Classificatiom 3B or above.<br>→Coating must provide with sufficient adhesion to the substrate and subsequent coating layers |
| Layer thickness | Rockhard Chromate primer has to be applied with a thickness of 15-35 µm. The thickness of the applied layer prerequisite for other key functionalities (e.g. corrosion resistance) | N/A |
| Chemical resistance | Resistance to all aircraft hydraulic, cooling, lubrication and fuel fluids.<br>Resistance to wet chemicals up to operating temperatures of 220 ºC. | **ISO 2812-1** or **MIL-C-22750**<br>**ISO 1518-1** or **ASTM D 7027 (Method B)** - dead load<br>→Coating must not show blistering or other film defects after 1 h in test conditions.<br>Primer after Chemical Immersion – must show no penetration under 1500 g minimum load when tested to ISO1518 or ASTM D7027. |
| Temperature resistance (thermal cycling resistance & thermal shock resistance) | Cycling: Resistance to all temperatures occurring during the service life of the individual component parts.<br>Shock: Resistance to prolonged exposure to high heat in dry air. Type and Batch* testing up to and including minimum 1000 hours resistance to 220ºC | Cycling: Temperature cycle between -55°C and 220°C with a 6 hour dwell at max. and min. temperature. Rate of temperature rise is not considered. Specimens are submitted for 25 cycles minimum.<br>→ No blistering or other defects attributable to the thermal exposure or failure of the coating. |
| Compatibility with substrate / other coatings | Compatibility with all metals with a primary concern for Al alloys, Mg alloys and steel alloys. | Tested according to BS EN ISO 2409. |
| Flexibility (Bend) | Flexibility describes the ability of the primer or overall coating to maintain its structure during the bending of the underlying substrate. | The coating shall not become detached or damaged |
| Scratch resistance | Coating system has to maintain its adhesion to the substrate and its cohesive integrity in the presence of impinging solid particles (such as sand or ice) or liquid droplets such as rain. | not applicable |

*Type & Batch testing: Type approval is required for all new products to be approved to the Rolls-Royce Material Specification. Alternatively, when a change in formulation of an existing approved product occurs. This may include a change in raw materials or raw materials supplier. All products produced in a single production run from the same batches of raw materials under the same fixed conditions, and presented for manufacturer's inspection at one time.

**Corrosion resistance**

Corrosion describes the process of oxidation of a metallic material due to chemical reactions with its surroundings, such as moisture derived from humidity and other sources, and which can create corrosive electrolytes through the presence of other dissolved substances. In this context, the parameter corrosion resistance means the ability of a metal to withstand gradual destruction by chemical reaction with its environment. For the aerospace industry, this parameter is one of the most important since meeting its minimum requirements plays a key role in assuring the longest possible life cycle of aerospace systems and all the implicit parts, the feasibility of repairing and maintenance activities and most importantly, continued safety and reliability of aerospace components during use. For example, the 2000-series aluminium alloys, commonly used in the aerospace industry, contain approximately 4 - 6 % of Copper (Cu) as an alloying element to provide the material strength. However, Cu as a noble element acts as built-in in corrosion driver. Inhibition of the Cu-induced corrosion is mandatory for long-term corrosion stability. The corrosion requirements vary within the aerospace industry and are dependent on the specific design parameters. As an initial screening step, corrosion of test panels or components is evaluated to failure per neutral salt spray tests using Cr(VI) formulations as a comparative control. This test can only be considered a very preliminary screening test that might be introduced in the early development process (e.g., TRL 2 as further discussed in chapter 5.3.1). When potential Cr(VI)-free candidates do not pass this test, there is no confidence that they will be able to meet the challenging performance requirements mandated by the aerospace industry. Even where such a test is successfully completed, extensive further testing over many years is required as a part of the pre-qualification process. In the aerospace industry, it is responsibility of the OEMs to qualify and approve alternatives as being fit for purpose, not the formulators. Aerospace companies follow a very robust validation plan to ensure safety. Changing a formulation needs to be substantiated and certified to ensure that the new formulation provides the necessary performance for the relevant design parameters.

Ideally, the corrosion-inhibiting substances / systems are applicable in all surface treatment processes, compatible with subsequent layers and perform effectively on all major metal substrates. Furthermore, it has to guarantee product stability (chemically and thermally) and has to reinforce the useful coating properties. Most importantly, for critical design parameters, the aerospace industry has set its systems-level performance standards and specifications for Cr(VI) replacements to reflect equivalency to Cr(VI) performance in order to maintain the industry’s very high and long-standing safety record.

**Active corrosion inhibition**

Active corrosion inhibition is the ability of a material to spontaneously arrest corrosion resulting from small amounts of chemical or mechanical damage (see Figure 8). This self-healing characteristic is essential because it reduces premature corrosion failures, enhances service life of parts, reduces maintenance intervals and improves flight security of air travellers and security of power supply. The active corrosion inhibition capability of pentazinc chromate octahydroxide-based surface treatments is seen in the positive corrosion test results of those coatings and is a key factor of the superior corrosion resistance of the Cr(VI) coatings seen in hardware already in-service. The lack of this capability makes it very difficult for Cr(VI)-free coating systems to perform equivalently. This characteristic is particularly important for systems with long service lives in harsh environments. Occasional abrasion and localized mechanical damage to surfaces sometimes occurs, compromising the protective layer.

**Adhesion promotion (adhesion to subsequent coatings or paint)**

Depending on the final functions of the treated component parts, they may be coated with additional layers (such as paint and adhesive films) and therefore adequate adhesion is essential for long-term performance. In this analysis, the parameter adhesion describes the tendency of particles or surfaces to adhere to one another. Regarding the aerospace industry, many parts are exposed to harsh environmental conditions, come into contact with other metallic parts or have to withstand strong mechanical forces. The requirements for adhesion vary within the aerospace industry and depend on the specific coating thickness and the function and location of the part. A variety of screening tests are used to evaluate coating adhesion. Even where such a test is successfully completed, extensive further testing is required to substantiate and certify that the new formulation provides the necessary performance for the relevant design parameters.

**Layer thickness**

The thickness of the different layers or coatings on the substrate (measured in microns) is crucial for the best performance of all parts of aerospace systems. The objective is to get maximum performance with minimum thickness which equates to weight. For example, weight is critical for fuel efficiency of an aircraft and has also economic relevance (e.g. number of passengers or amount of luggage per passenger). Not meeting the specified requirements of this parameter could lead to deficiencies in other characteristics of the pieces, for example reduced corrosion and chemical resistance, improper adhesion of coatings to the substrate or decreased cracking resistance. The process capabilities of Cr(VI)-free primer systems need to be considered in order to guarantee even coverage of three-dimensional geometries. Film thickness is tightly controlled by company and industry specifications.

The specification requirement for the stoved epoxy primer with pentazinc chromate octahydroxide (Rockhard Chromate primer) is 15 - 35 microns (µm) depending on the engine component part, its functionality and its individual exposure to mechanical and physical-chemical loads.

**Chemical resistance**

This parameter is defined as the ability of solid materials, in this case cured primer, to resist damage by chemical exposure. Aerospace components are often exposed to jet fuel, hot oil, hydraulic fluid, and other corrosive chemicals. Consequently, the potential Cr(VI)-free alternative coating layer must be unaffected by prolonged exposure to these materials during use. Fuel and hot oil immersion tests called out in specifications are tools for screening suitability of proposed alternative compositions. Any suitable candidate alternative coating must provide the necessary performance for the relevant design parameters.

**Temperature resistance - thermal cycling resistance and thermal shock resistance)**

The parameter thermal cycling resistance describes the ability of a primer to withstand repeated low and high temperature cycling. For the same reasons stated above, it is vital that parts and coatings are able to perform their functions optimally at all temperatures to which the parts are going to be exposed during their service life. In general, different methods are available within the aerospace industry, where aerospace components have to meet test requirements to operate at both, very cold (< -50°C) and extremely high (> +200°C) temperatures. Thermal cycling requirements are tightly controlled by company and industry specifications.

Furthermore, the parameter thermal shock resistance describes the ability of the primer to withstand prolonged exposure to heat in dry air conditions without blistering or other defects attributable to the temperature load.

**Compatibility with substrates / other coatings**

Compatibility with a wide range of substrates (primarily Al-, Mg- and steel-alloys) and other coating components (i.e. other primers or paint) is a key performance characteristic within the aerospace industry. To determine the compatibility with substrates or topcoats, adhesion testing is carried out according to company and industry specification requirements (see section **Adhesion promotion**).

**Flexibility (Bend)**

The parameter flexibility describes the ability of the primer or overall coating to maintain its structure during the bending of the underlying substrate. Aircraft engine component parts, made of different substrates, are not rigid but are built to allow a certain degree of movement to prevent stress (i.e. friction) during operation. Additionally, due to the thermal expansion of the substrate occurring under load, a primer or the overall coating has to be able to expand to the same degree as the substrate as otherwise primer / coating detachment or damage would result.

**Scratch resistance**

This parameter describes the ability of a coating system to maintain its adhesion to the substrate and its cohesive integrity in the presence of impinging solid particles (such as sand or ice) or liquid droplets such as rain. Scratch resistance is a critical property for coatings that are exposed to high speed air streams. Scratch resistance requirements are tightly controlled by company and industry specifications.

## 4. ANNUAL TONNAGE

The annual tonnage for the use of pentazinc chromate octahydroxide is 0.06 tonnes / year.

# 5. IDENTIFICATION OF POSSIBLE ALTERNATIVES

## 5.1. List of possible alternatives

IDP Ltd. conducted extensive R&D on Cr(VI)-free corrosion inhibitors for the substitution of pentazinc chromate octahydroxide used in the product Rockhard Chromate primer. Several Cr(VI)-free corrosion inhibitor compounds were examined either alone or in mixtures. The following Table 7 provides an overview on all compounds / mixtures which have been taken in consideration as potential Cr(VI)-free substitutes by IDP Ltd. and which have been evaluated by RR in the respective Cr(VI)-free primer formulations. The alternatives have been tested to the requirements of the RR Material Specifications and were dismissed due to insufficient corrosion protection. This list is a combination of suggestions from raw material suppliers, the results from extensive R&D work and suggestions from industry related literature.

**Table 7: Cr(VI)-free corrosion inhibitors and technical limitations (exclusion criteria)**

| Cr(VI)-free corrosion inhibitor (CAS no.) | Technical Limitations |
|---|---|
| Magnesium hydrogen phosphate trihydrate (7782-75-4) | Insufficient corrosion protection |
| Titanium oxide (TiO1.7) (66402-68-4) | Insufficient corrosion protection |
| Zinc phosphate (7779-90-0) / zinc molybdate (13767-32-3) | Insufficient corrosion protection |
| Aluminium dihydrogen triphosphate (13939-25-8) | Insufficient corrosion protection |
| Strontium hydrogen phosphate (13450-99-2) / calcium silicate (13983-17-0) | Insufficient corrosion protection |
| Strontium hydrogen phosphate (13450-99-2) | Insufficient corrosion protection |
| Silicon dioxide (7631-86-9) | Insufficient corrosion protection |
| Zinc oxide (1314-13-2) | Insufficient corrosion protection |
| Zinc phosphate (7779-90-0) / Zinc oxide (1314-13-2) | Insufficient corrosion protection |
| Zinc-5-nitroisophtalate (60580-61-2) | Insufficient corrosion protection |
| Benzotriazole (95-14-7) | Insufficient corrosion protection |
| Methyl-1H-benzotriazole (29385-43-1) | Insufficient corrosion protection |

The base of the Rockhard Chromate primer is a thermally cross linked resin system. Extensive R&D testing showed that the performance of the Cr(VI)-free anti-corrosive additives is affected by the thermal cure cycle. IDP Ltd.'s R&D work has included a comparison of individual use and combinations of these compounds. Due to the required thermal curing process, it is not possible to transfer the results from Cr(VI)-free cold cured two-pack epoxy primer systems to the stoved Rockhard Chromate primer. This means that a Cr(VI)-free solution, which has proven its function in a primer system A, does not necessarily provide the same functionality in a differently applied primer system B (in this case: cold-cure two-pack epoxy primer versus stoved epoxy primer system).

To date none of these compounds or mixtures are able to meet the high corrosion requirements set out by RR. The performance weakness of the most important key functionality corrosion resistance is observed during hot salt spray, and cyclic corrosion testing. The testing conditions are described in chapter 3.3.3. In both instances, the Cr(VI)-free anti-corrosives fail to prevent pitting corrosion of Al alloys. If corrosion protection on Al alloys cannot be proven, Mg alloys are not tested as these are much more susceptible for corrosion.

**Please note that testing for corrosion resistance is the first screening to evaluate the potential of an Cr(VI)-free alternative as this is the most important key functionality of the primer. If the test and specification requirements described in chapter 3.3.3 are not met, no further testing is conducted with this potential substitute. However, testing on other key functionalities described in chapter 3.3.3 is initiated in parallel to safe R&D time, if the Cr(VI)-free alternative has proven sufficient corrosion inhibiting properties.**

## 5.2. Shortlist of alternatives

During the HITEA project (2013 - 2015), three formulations proved to be suitable for further investigation. These formulations are in focus of current R&D activities on the substitution of pentazinc chromate octahydroxide.

In general, the procedure for the development of a Cr(VI)-free primer formulation includes primarily the substitution of the chromate compound for a Cr(VI)-free alternative. The base composition of the primer system, including resin, catalysts, solvents, etc., remains mostly the same as it is well-established and key functionalities such as chemical and heat resistance, adhesion, compatibility with substrates / other coatings are highly dependent on those components. Of course, formulation adjustments (e.g. additives) are made step by step for further improvement of the overall primer system. A graphical presentation on the R&D efforts with respective timeline and information on the tested primer formulations is provided in Figure 9 down below.

Please note that some primer formulations evaluated below were developed by J&L's primer business unit, which became part of IDP Ltd. in the meantime.

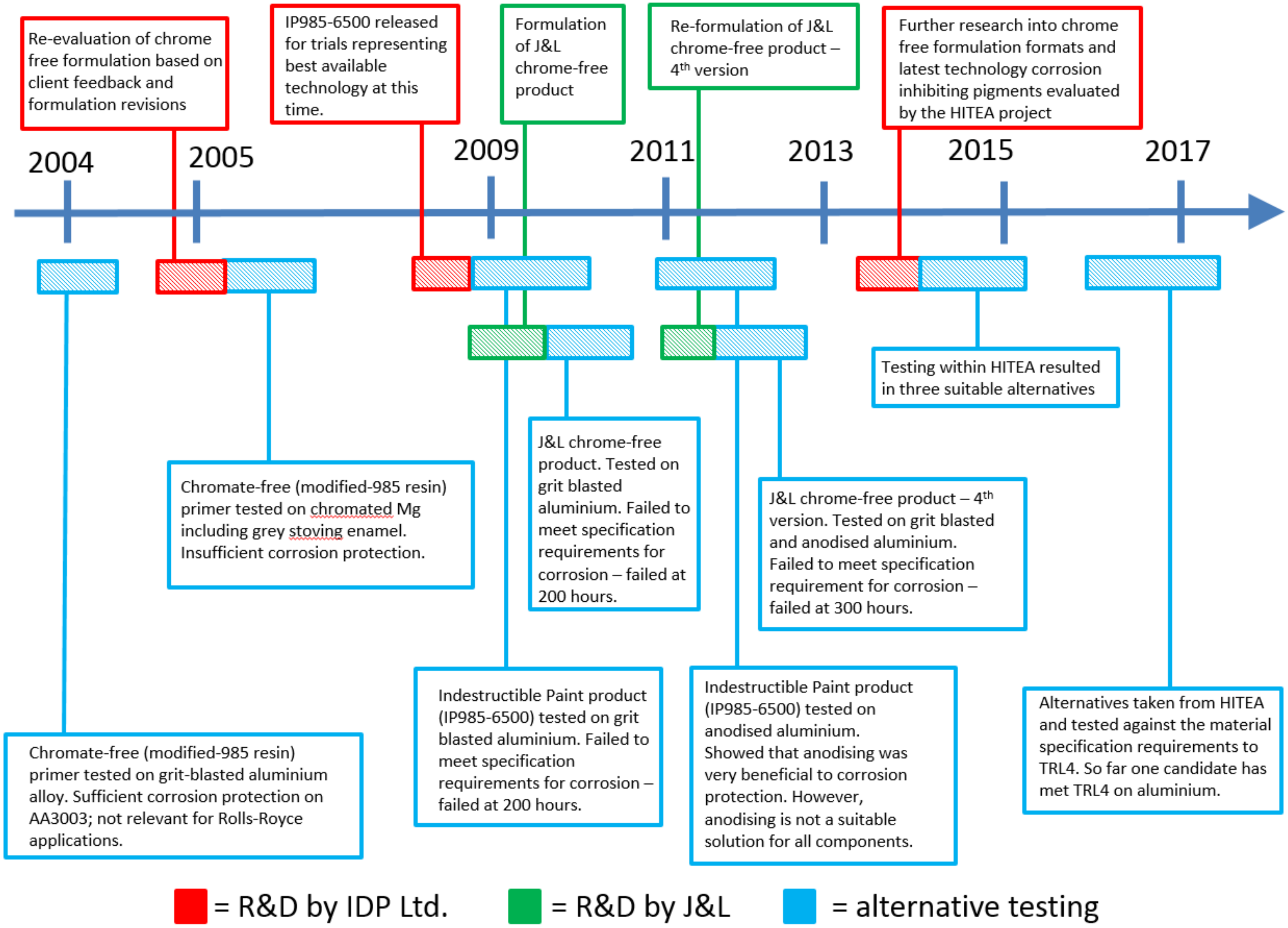


**Figure 9: Graphical presentation of the R&D timeline for the development of an alternative primer formulation**

Ongoing substitution efforts and technical performance for these proprietary formulations will be discussed in more detail in chapter 5.3.2 and chapter 6.

To further highlight the difficulty of the substitution of pentazinc chromate octahydroxide in Rockhard Chromate primer is should also be re-emphasised that IDP Ltd. does **not** itself conduct R&D on new corrosion inhibiting substances but rather uses chemistry developed by other specialist organisations for their respective R&D on new primer formulations. The formulator has to track developments through contact with these specialist organisations and keeping up to date with published research to identify and seek to incorporate new candidates in its own R&D efforts to formulate novel coatings. Therefore, the R&D work is also highly dependent on the market availability of these substances.

## 5.3. Description of efforts made to identify possible alternatives

The preparation of this AoA has been conducted by close cooperation of IDP Ltd., the formulator of the Rockhard Chromate primer containing pentazinc chromate octahydroxide and the OEM RR in the value chain of this specific product.

**For specific information on the efforts made up to date by both, formulator and OEM, to find a suitable alternative for pentazinc chromate octahydroxide in Rockhard Chromate primer please refer to chapter 5.1 and chapter 5.2.**

### 5.3.1. Research and development

As mentioned earlier in this document, a large amount of commissioned research over the last 25 years (since 1992) pursued identification and development of viable alternatives to chromates such as pentazinc chromate octahydroxide. The unique functionalities of Cr(VI) compounds (explained in detail in chapter 3.3.2) make it challenging and complex to replace the substance in surface treatment processes where superior corrosion protection or adhesion properties are required to ensure safe performance in demanding environments. Aerospace engine components are complex systems involving not only design of the device, but also its use and maintenance history in varied climates and service. An aircraft engine is exposed to massive forces and extremely high stress levels due to high velocities and environmental impacts. Therefore, every part is designed and manufactured with consideration to system and component interactions. In a complex system, change introduces new forms of risks and uncertainties that can lead to anticipated and unanticipated failures.

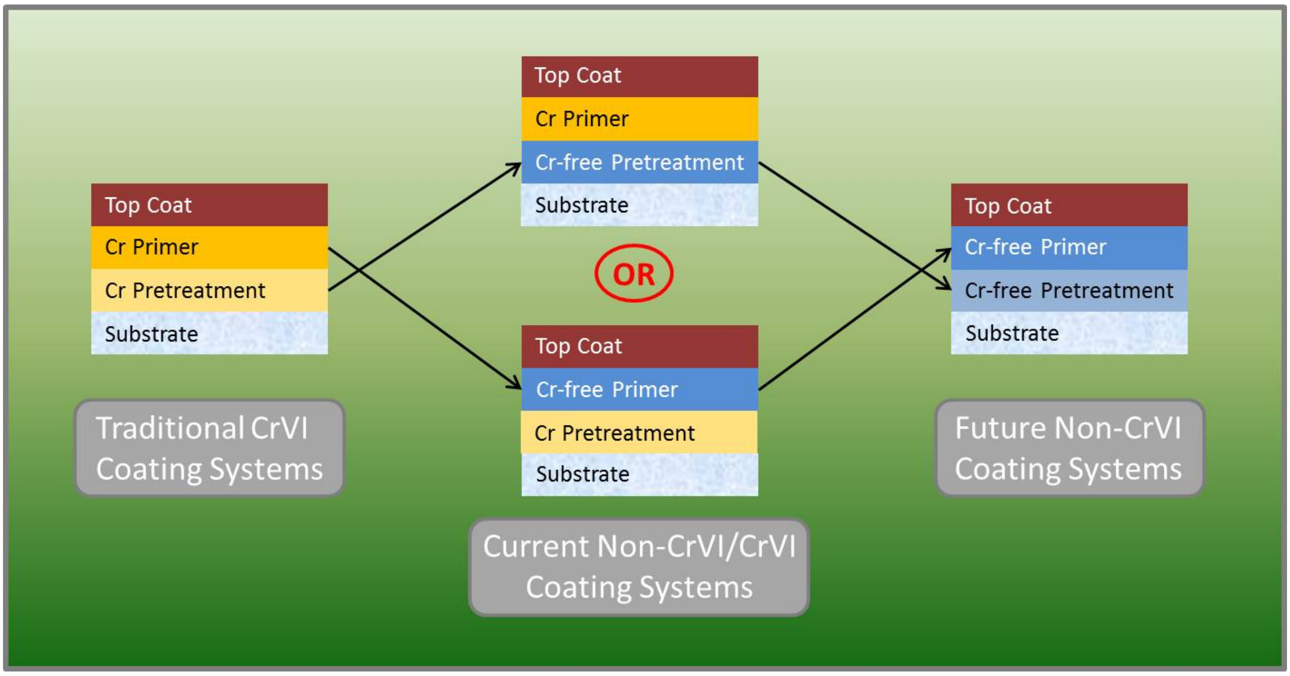


**Figure 10: Development of coating systems - from the past to the future (AMMTIAC 2012)**

Decades of service has built confidence in chromates performance for corrosion protection and other parameters across many differing technologies. This knowledge facilitates and enables exact lifetime predictions for component parts and therefore has significant influence on the level of risk created though substitution with alternative chemistries in safety-critical aerospace engines. Therefore, to minimise risk, a rigorous approach to research and design of alternative technologies is needed. A typical scheme for the development of Cr(VI)-free multi-layer coatings (e.g. primer or pre-treatment) is illustrated in Figure 10.

Development of Cr(VI)-free multi-layer coatings may be considered when fundamental new design explores the use of a different substrate with much lower potential for corrosion. In practice, such new designs are often technically or economically unfeasible.

In broad terms, the substitution of the REACH affected technology, can be broken down into three distinct phases: development / feasibility, validation / qualification and certification / industrialisation / deployment, as seen in Figure 11. Close alignment exists between each of these phases and TRLs, as described in Annex A. New technologies must pass the criteria of each phase before they can proceed to the next. If a technology does not pass the criteria, then it is discontinued as a candidate.


**Figure 11: Typical phases of technology development within the Aerospace industry (Rolls Royce, 2016).**

TRLs are a gated method of estimating new technology maturity during the qualification, validation, and certification process. TRLs are based on a scale from 0 to 9 with 9 being the most mature technology.

- TRL0 through TRL4 are development levels where feasibility is assessed;
- TRL5 through TRL6 are validation / qualification levels;
- TRL7 through TRL9 are the certification / industrialisation / deployment levels.

To obtain design approval, a process requires TRL6 as a minimum. Design approval is the first level at which a potential Cr(VI)-free alternative to pentazinc chromate octahydroxide would be considered for use in aerospace engine components. TRL testing is not for a substance or coating but rather a substance or coating system in specific design parameters (meaning that if you want to use the same coating in different design parameters, you must repeat the TRL process for each of the design parameters). TRL 6 requires testing in a 'relevant environment', which could include an engine or flight test.

Cr(VI) formulations are very versatile from a design parameter perspective due to their wide range of key functionalities and sustained performance history. Many times during analysis and comparison of alternatives to Cr(VI), favourable, previously unidentified attributes or properties of the chromate compound are identified. This leads to additional performance criteria that the potential alternative must meet to match the performance of Cr(VI) for those design parameters. Significant effort is required to develop and execute testing that validates candidate alternatives to pentazinc chromate octahydroxide according to the TRL process. Determining the extent of the testing required to implement a new or alternative technology is on a case-by-case basis due to the many design parameters considered in order to quantify the risk of corrosion. These include but are not limited to:

1. Design of the part or assembly (e.g. substrate, inclusion or proximity to dissimilar materials or mating surfaces, crevices that can entrap liquids, structural stress and strain environment, etc.)
2. Environmental conditions within the product (e.g. location, presence of condensation or liquids, entrapment of liquids, temperature range, microbial growth, etc.)
3. External environmental conditions (humidity, wind / rain erosion, impact from runways, exposure to fluids like de-icers and hydraulic fluids, etc.)
4. Probability of finish deterioration during use (e.g. chipping, scratches, abrasion, erosion)
5. Historical performance in similar aerospace applications
6. Previous issues due to variation in maintenance practices
7. Ability to inspect during the lifetime of the product

These principles have been developed over the lifetime of many products and continue to be refined based upon service related performance and experience with new materials, processes and designs. All of the above affect performance and must be considered and evaluated before an alternative is approved for use. For each single part, OEMs determine precise technical performance requirements and include these requirements in the manuals. These requirements can vary significantly between engine or aircraft models. Adherence to the technical requirements ensures candidate performance and safe operation of aerospace engine systems. Therefore, the requirements always reflect system-level requirements (e.g. airworthiness regulations).

A screening process utilizing standard test methods evaluates performance parameters with respect to corrosion resistance of primers (see chapter 3.3.3). Besides requirements for the production of the original part, additional specifications are defined for MRO activities, which are in the OEM manuals as well.

Implementation depends on the scale of the modification to the existing, proven design and its relevant requirements. In any case, it takes considerable effort and time to establish the specific testing requirements and funding for an alternative corrosion inhibitor or design (system or component). It will take many more years to carry out the testing; the necessary testing cannot be accelerated, as extrapolation from limited data is unlikely to be accepted as reliable.

In the early development process (e.g. TRL0 and TRL1), much of the research effort was conducted by the formulators who carried out initial feasibility studies to assess the viability of potential alternatives before advising the OEMs of candidates that might be considered for more extensive assessment. This process in itself requires a substantial effort. A primer formulator reported that efforts to develop alternative formulations have been underway for over 30 years. In addition, for every candidate alternative released to the OEMs for further testing, over 100 formulations were tested and eliminated by the formulator due to technical failures. As a result, the number of candidate alternatives released to the OEMs by this formulator has been relatively small due to the challenge of replacing strontium chromate. While a formulator does preliminary assessment of the viability of a potential alternative, only the design owner can determine when a candidate alternative is fully validated and certified for each of their uses of the candidate formulation.

Once at the OEM, each candidate alternative goes through a process based on TRL methodology, beginning with initial screening testing (TRL2 to TRL3). The goal for initial screening tests is to assess candidate performance against a broad range of design parameters (e.g., alloys, temperature exposures, operational conditions). As failures occur (e.g., unacceptable corrosion on specific alloys), a risk analysis is performed and a decision is made to either narrow the subset of design parameters or end testing entirely. This decision process is repeated in further qualification testing (TRL3 to TRL6), ultimately identifying the scope of applicability, if any. As shown in Figure 12, a candidate alternative may only be industrialised for a subset of the original goal (in some cases a single part in a specific engine or aircraft model). Niche design parameters / applications refers to this narrowed scope of applicability.

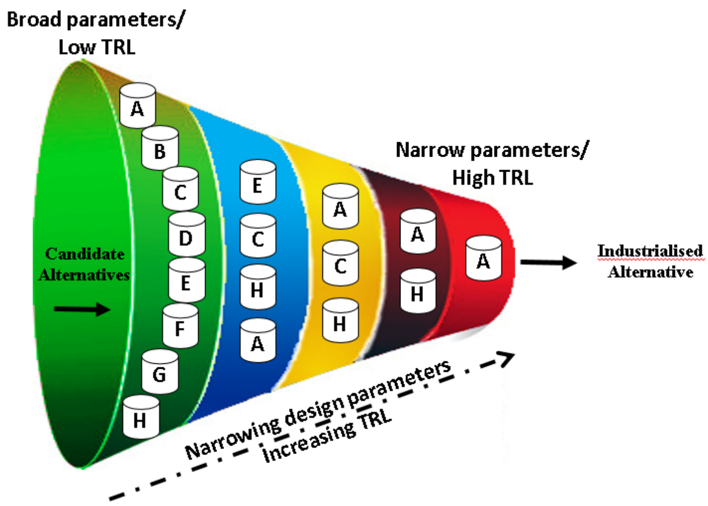


Figure 12: Schematic representation of screening process for Candidate Alternatives (A through H) with respect to design parameters and TRL level.

In addition to efforts carried out by individual companies, industry research consortia are actively assessing new technologies (TRL1 to TRL3). Industry collaborations have the advantage of sharing development costs and knowledge of non-competitive technologies under a legal framework. However, individual companies and their supply chains must perform higher TRL validation and deployment of these new technologies (TRL4 to TRL9). Pass criteria for technologies across all TRLs can be aligned to industry and internal company standards.

Please refer to Annex A for detailed information on the individual contents of the TRL phases.

Further challenges exist for legacy aerospace components. Production, maintenance, and repair of in-service aerospace systems must use certified materials and processes. When in the early design phase for new aerospace systems, there is an opportunity to consider introducing a material change. Significant design changes are seldom introduced into in-service aerospace systems, as this could involve substantial technical risk and cost. The more complex a system, the more likely it is that small changes can have significant unforeseen impact. Thus changing a single detail of a part in an aerospace system is highly complex, time consuming, and can lead to unanticipated failures and repercussions. Systematic TRL-style implementation helps to minimise the impact of these unanticipated failures and repercussions.

Existing legacy designs are not able to take advantage of a new product development cycle to validate and incorporate these technologies. Thus, legacy designs require a new testing and substantiation plan to ensure, at a minimum, the same level of performance exists. In order to approve new aerospace system models, costly and time-consuming engine and flight tests are required as a part of the validation / certification process. To qualify candidate alternatives on in-service models, these tests must also be performed. Complex design factors, detailed analyses, and product testing impede incorporation of an alternative base material into existing legacy designs.

**Indestructible Paint / Rolls Royce Specific and Key Collaborative Research Programmes**

Hexavalent chromium substitution work in the aerospace industry has been ongoing for 30+ years, and has focused on replacing Cr(VI) processes, where feasible. This work, however, has not specifically been tracked at a substance level.

Aerospace companies assess many factors when prioritizing substitution work, including, but not limited to:

1. Review of all relevant processes which use Cr(VI)
2. Determination of the availability of candidate alternatives for these processes.
3. Bench marking of formulators and other aerospace companies' successes and failures with testing potential and candidate alternatives.

4. Identification of the number of parts that use the coating / processes that could potentially benefit from this substitute.

The OEMs will review the above information, and prioritise their replacement tasks based on the information they have gathered. The status of the candidate alternatives and their perceived likelihood of successfully meeting the OEM's design requirements is often a critical factor in the decision, as is the hazard assessment of the alternatives.

Rolls-Royce has a continuing programme seeking to substitute Cr(VI) used within materials and manufacturing processes. The business continuity risk created by chemical substance regulations, such as REACH, has increased the importance of this programme and there has been recent success in substituting Cr(VI) from a number of manufacturing processes.

The REACh Compliant Hexavalent Chrome Replacement for Corrosion Protection, HITEA (Highly Innovative Technology Enablers for Aerospace) funded project was initiated in 2012; a 17-member consortium was formed consisting of aerospace OEMs (including Rolls Royce), suppliers, formulators and academics with the goal to identify and evaluate suitable alternative systems. At the end of the project, over 45 candidates were tested in over 160 systems and there were no equivalent one-to-one alternatives for the main aerospace applications that were being assessed. However, there were candidates that delivered some of the required properties for specific design parameters. Further development and validation of these candidates is required before they can be deployed onto aerospace products, which may take up to another 10 years. Moreover, significant development is required for the design parameters where no suitable alternatives were identified.

The International Aerospace Environmental Group (IAEG) Working Group 2 (Replacement Technologies) has been working on several fronts to address environmental and chemical regulations facing the aerospace industry since its formation in 2011. A number of companies in GCCA are also active in this forum, including Rolls-Royce. One of the IAEG efforts involves working through its member companies to conduct research to pursue the development and implementation of hazardous chemical alternatives throughout the value stream that meet aerospace performance and safety requirements. This effort has focused on Cr(VI) free alternatives for passivation of corrosion resistant steel and anodising of aluminium parts – including anodise seal. In addition, there are two Cr(VI)-free primer projects which are being launched in the near future. The aim of these projects is to share available information on alternatives in order to reduce cost and risk of testing within companies who are behind the curve with their substitution activities.

Additionally, IDP Ltd. is also part of two currently running research programs, SOLMAG and COLM (Corrosion on Light Metals) project, for further R&D on Cr(VI)-free primer formulations.

The intention of the SOLMAG project is to advance the industrial use of a novel Sol-Gel technology that was successfully demonstrated by Sheffield Hallam University and IDP Ltd. during a funded feasibility study. The objective is to realise the manufacture of small scale industrial volumes of the Sol-Gel for aerospace applications.

In May 2016, eight companies and two universities established a research consortium, partially funded by Innovate UK, the UK's Innovation agency. The key objective is to develop and industrialise safe alternatives to chromate conversion coatings that are used for aluminium components within the aerospace and defence industries. Additionally, the project also demonstrates the continuing commitment by the industry to remove potentially hazardous substances from its manufacturing processes, OEM products and MRO activities. The three year project was bid for within the Highly Innovative Technology Enablers in Aerospace (HITEA 3) competition and the research consortium is called Advanced Hex Chrome-free Surface Technologies for Corrosion Protection (TSB file no. 102580). However, COLM (Corrosion of Light Metals) has been adopted for simplification.

### 5.3.2. Substitution efforts related to pentazinc chromate octahydroxide

As indicated above, R&D on substitution of pentazinc chromate octahydroxide started in 2004. It took more than 10 years until 2015 to identify potential candidates that might be suitable as alternative (see Figure 9). After extensive screening tests were performed to evaluate initial feasibility, it took approximately 13 years to develop the candidate alternatives to the point where TRL4 is partly met (depending on formulation tested and alloy). Currently, performance is assessed against a broad range of design parameters (e.g. different alloys, temperature exposures, operational conditions). However, the current formulations do not provide the required corrosion protection on all required alloys as described in the following chapter. Re-formulation will need to be carried out to enhance the corrosion protective properties.

As next steps, decision process under which conditions and design parameters the performance of each candidate could be sufficient will be repeated in further qualification testing, ultimately identifying the scope of applicability for each formulation, if any (TRL6). From previous experience, it is estimated that it will take at least 8 years to progress through TRL 6. It is important to note that success through this crucial R&D stage is not assured and alternative failure can occur at any time, which would obviously lead to extended R&D time periods. Until TRL9, subsequent evaluation of the alternative on all necessary component parts, at least another 6 years are required.

**Please note that the time schedule of 6 years for passing TRL 9 is only a best case estimate. From experience with other projects this evaluation phase can also easily require more than 7 years.**

Concluding, based on the time periods stated by IDP Ltd., a review period for the continued use of pentazinc chromate octahydroxide in Rockhard Chromate primer of **at least 12 years** is required to ensure the development of an unequivocally and absolutely safe use of the substitute in safety critical aerospace engine components. For a graphical derivation of the review period please refer to Figure 13.

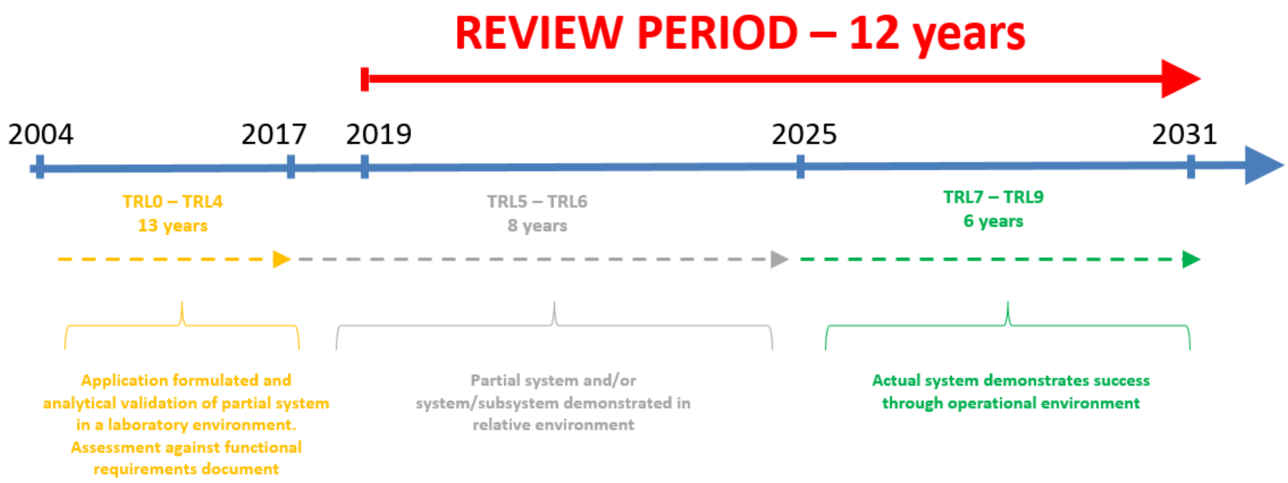


**Figure 13: R&D timeline for substitution of pentazinc chromate octahydroxide in Rockhard Chromate primer and derivation of the length of the review period**

### 5.3.3. Data searches

IDP Ltd. is part of the REACH compliant Hexavalent Chrome Replacement for Corrosion Protection (Highly Innovative Technology Enablers for Aerospace (HITEA) project. In the first place, searches for publically available documents were conducted to ensure that all potential Cr(VI)-free corrosion inhibitors have been taken in consideration for the replacement of pentazinc chromate containing Rockhard Chromate primer in aerospace and aeroderivative applications.

Furthermore, the testing of the most promising and recent Cr(VI) candidates was conducted under the consortium (HITEA project) from beginning of 2013 to mid 2015. The HITEA project found **three** candidates to be suitable for the substitution of pentazinc chromate octahyroxide in Rockhard Chromate primer. Rolls Royce, as user of the Rockhard Chromate primer on aircraft engine components for aerospace and aeroderivative applications, have tested the alternative Cr(VI) candidates and the respective corrosion result reports are available to IDP Ltd.

Additionally, IDP Ltd. is also part of two currently running research programs, MAGSOL and COLM (Corrosion on Light Metals) project, for further R&D on Cr(VI)-free primer formulations.

### 5.3.4. Consultations

A scoping meeting was carried out with the relevant companies formulator and aircraft manufacturer from the aviation sector) for definition of the concrete function of the primer, discussion of the scope, overview on R&D efforts and the current state of experience with alternatives. Prioritisation of critical parameters and the minimum technical requirements were discussed.

At this stage of the data analysis, information on previous R&D activities was provided showing that several alternative corrosion inhibitors had been screened out as they failed to pass the corrosion requirements (see Table 7).

Discussions with technical experts followed by a final data analysis led to the formation of a shortlist of **three** candidate alternatives that are currently under R&D for the applications in scope of this AfA. These candidate alternatives are assessed in detail in the following chapter.

Over the last years, several chrome-free products were placed on the market, while the quantity of Cr(VI)-containing products remained stable. However, this is also related to the increased production and demand of aircraft and other aeronautic products over the last years and with a tendency to further increase.

Most importantly, availability on the market does not allow drawing any conclusions on the technical maturity of these products to replace the Cr(VI)-based solutions for aeronautic applications. Generally, all material and processes incorporated in an aircraft must be qualified, certified and industrialised by the production certificate holder as described in detail in Annex A.

**For the use applied for, there are no chromate free alternatives available and approved which meet the specifications from the applying companies from the aviation sector.**

# 6. SUITABILITY AND AVAILABILITY OF POSSIBLE ALTERNATIVES

The three alternatives assessed in this chapter are considered the most promising, where considerable R&D efforts have been carried out within IDP Ltd. They ether show technical limitations when it comes to the demanding corrosion requirements for aerospace applications and / or are not mature enough to be used as drop-in alternative to Rockhard Chromate primer containing pentazinc chromate octahydroxide.

To assess the technical feasibility of the alternatives, colour coded summary tables are included in the document. The colours are as follows:

| Colour | Explanation |
|---|---|
| (red) | Not sufficient - the parameters/assessment criteria do not fulfil the requirements |
| (yellow) | Requirements partly met |
| (green) | Sufficient - the parameters/assessment criteria do fulfil the requirements |
| | No data available |

**Importantly, a potential Cr(VI)-free alternative must fulfil all key functionalities for aerospace applications to successfully substitute pentazinc chromate octahydroxide in Rockhard Chromate primer.**

## 6.1. ALTERNATIVE 1 – LR2992 primer

### 6.1.1. Substance ID and properties

The exact primer formulation is proprietary to IDP Ltd. However, the corrosion inhibiting substance used is [redacted].

The candidate is tested on Al-, Mg- and steel alloys at laboratory scale.

### 6.1.2. Technical feasibility

The LR2992 candidate formulation in principle provides the performance required by the RR Material Specifications. Currently, performance testing is ongoing to evaluate the equivalency to the benchmark material, pentazinc chromate octahydroxide. However, proof of concept is only given in laboratory application on Al- and steel alloys, which corresponds to TRL4. The LR2992 primer formulation is currently not mature enough to also prove the required corrosion performance on Mg alloys.

Therefore, further testing is required to develop the concept as part of component, system, sub-system or prototype in the relevant environmental conditions. All alternative corrosion inhibitors require also a change in their respective formulation (e.g. resin, catalysts, etc.), which then makes full material testing necessary to evaluate the chemical, heat, and mechanical integrity of the coating alongside the corrosion protection properties.

*Corrosion resistance:* Sufficient corrosion protection has not been achieved yet for all substrates which are subject to this AoA. The LR2992 ([redacted]) has passed the TRL4 requirements only for aluminium alloys. However, there is still testing to be completed on magnesium. And a residual question over the requirements on steel. Even the benchmark system exhibited issues on the corrosion resistant steel. Historically for qualification, where a product (paint) did not meet the requirements on mild steel the test substrate has been changed to corrosion resistant steel. This issue typically needs to be investigated separately. Therefore, it is becoming increasingly clear that a like for like replacement of the Cr(VI) containing coating systems is not applicable.

Please note that corrosion resistance is the most important functionality of a primer used for engine component parts in aerospace and aeroderivative applications. Therefore, during the assessment of an alternative, corrosion resistance is always tested in the beginning. Testing on all other key functionalities is initiated in parallel to the corrosion tests to safe R&D time if the alternative formulation shows sufficient corrosion performance.

**Table 8: Colour-coded assessment of the candidate primer LR 2992**

| Corrosion resistance | Adhesion | Layer thickness | Chemical resistance | Temperature resistance | Compatibility (substrate / coating) | Flexibility | Scratch resistance |
|---|---|---|---|---|---|---|---|
| | | | | | | | |

### 6.1.3. Economic feasibility

Against the background that the alternative formulation has not reached the technical readiness to be used as an alternative to Rockhard Chromate primer containing pentazinc chromate octahydroxide, no detailed analysis of economic feasibility was conducted. With flight safety at stake, it is the technical performance of the alternative first and foremost must be proven (i.e., equal or better performance to what is being replaced). If technical feasibility of the alternative is assured, then economic feasibility is assessed for further input into the business implementation plan (that is non-capital and/or capital resources).

### 6.1.4. Reduction of overall risk due to transition to the alternative

[redacted]

[redacted]

### 6.1.5. Availability

Current investigations are in TRL4. As described in chapter 5.3.2, it is estimated that until implementation of the alternative on Al alloys a review period of at least 12 years is needed. For the implementation on Mg- and steel alloys additional more time will be required as TRL4 has not been reached for these substrates.

### 6.1.6. Conclusion on suitability and availability for Alternative 1

Although the technical performance of this formulation is promising, its hazard profile creates doubts if this should be considered as a candidate of choice. However, as this candidate just recently passed TRL4 for Al alloys but not for Mg- and steel alloys, a review period of **at least 12 years** would be needed (even for the Al alloys as depicted in Figure 14) until full development and implementation could be reached. For the derivation of the review period please refer to chapter 5.3.2.

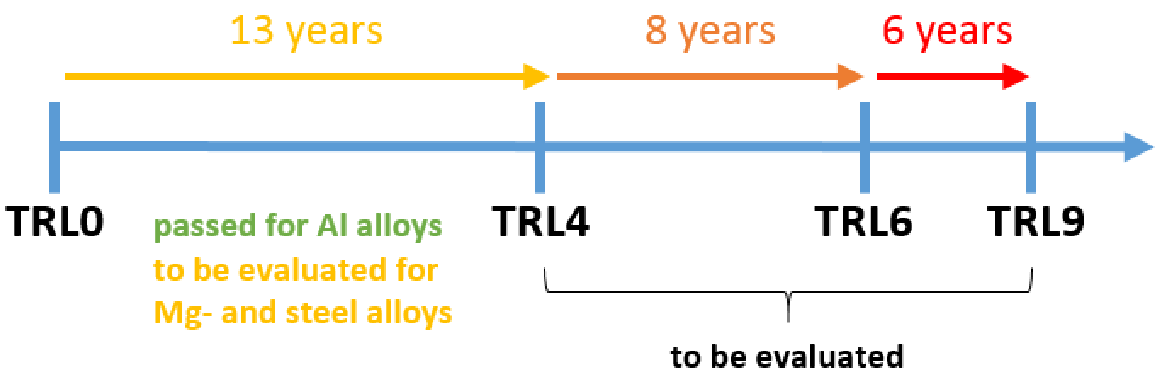


**Figure 14: Current R&D stage of LR2992 primer formulation**

## 6.2. ALTERNATIVE 2 – LR2965 primer

### 6.2.1. Substance ID and properties

The exact primer formulation is proprietary to IDP Ltd. [redacted].

The candidate was tested on Al- and steel alloys at laboratory scale.

### 6.2.2. Technical feasibility

The LR2965 candidate formulation does not provide the technical performance required by the RR Material Specifications to pass TRL4. The Cr(VI)-free formulation failed in the most important and critical key functionality corrosion resistance. Performance was not given in the Neutral Salt Spray test according to **ASTM B117** exemplarily depicted in Figure 15 below. [redacted]

[redacted]

**Figure 15:** [redacted]

Table 9. Colour-coded assessment of the candidate primer LR2965

| Corrosion resistance | Adhesion | Layer thickness | Chemical resistance | Temperature resistance | Compatibility (substrate / coating) | Flexibility | Scratch resistance |
|---|---|---|---|---|---|---|---|
| | | | | | | | |

### 6.2.3. Economic feasibility

Because of the technical failure of the Cr(VI)-free alternative, no quantitative and detailed analysis of economic feasibility was conducted. With flight safety at stake, it is the technical performance of the alternative first and foremost must be proven (i.e., equal or better performance to what is being replaced). If technical feasibility of the alternative is assured, then economic feasibility is assessed for further input into the business implementation plan (that is non-capital and/or capital resources).

### 6.2.4. Reduction of overall risk due to transition to the alternative

[redacted]

### 6.2.5. Availability

The current LR2965 primer formulation failed to pass TRL 4, mainly due to insufficient corrosion performance during the Neutral Salt Spray test. Accordingly, identification of root cause failure will be carried out and afterwards re-formulation is necessary. As described in chapter 5.3.2, the development of this primer formulation to TRL9 will required a review period of significantly **more than 12 years**.

### 6.2.6. Conclusion on suitability and availability for Alternative 2

Due to the fact that technical performance in the most important and critical key functionality corrosion resistance is not given, the Cr(VI)-free primer formulation cannot be regarded a suitable alternative for Rockhard Chromate primer containing pentazinc chromate octahydroxide in safety critical aerospace and aeroderivative applications. The technical performance criteria for passing TRL4 were not met by the alternative (see Figure 16).

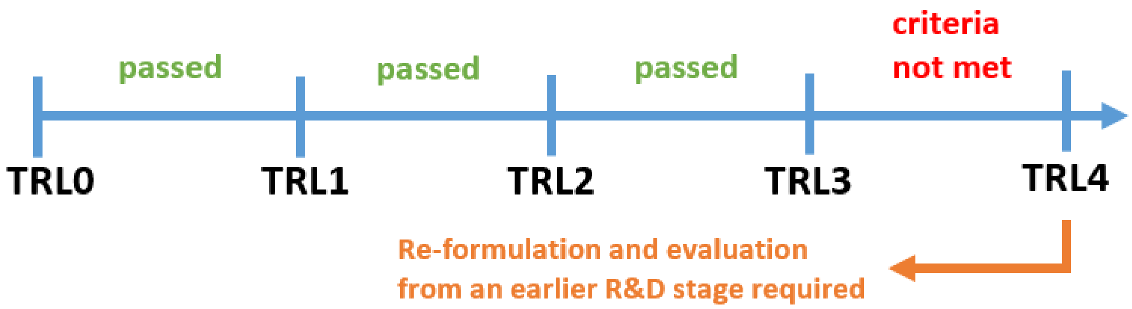


**Figure 16: Current R&D stage of LR2965 primer**

## 6.3. ALTERNATIVE 3 – LR2964 primer

### 6.3.1. Substance ID and properties

The exact primer formulation is proprietary to IDP Ltd. [redacted]

The candidate was tested on Al- and steel alloys at laboratory scale.

### 6.3.2. Technical feasibility

The LR2964 candidate formulation does not provide the technical performance required by the RR Material Specifications to pass TRL4. The Cr(VI)-free formulation failed in the most important and critical key functionality corrosion resistance. Performance was not given in the Cyclic Heat / Salt Spray test (2 hr at 220 °C, 20 hr in salt spray to **ASTM B 117** or **BS EN ISO 9227)** nor in the Neutral Salt Spray test according to **ASTM B117**.

**Table 10. Colour-coded assessment of the candidate primer LR2964**

| Corrosion resistance | Adhesion | Layer thickness | Chemical resistance | Temperature resistance | Compatibility (substrate / coating) | Flexibility | Scratch resistance |
|---|---|---|---|---|---|---|---|
| | | | | | | | |

### 6.3.3. Economic feasibility

Against the background that the alternative formulation did not prove to be suitable, no detailed analysis of economic feasibility was conducted. With flight safety at stake, it is the technical performance of the alternative first and foremost must be proven (i.e., equal or better performance to what is being replaced). If technical feasibility of the alternative is assured, then economic feasibility is assessed for further input into the business implementation plan (that is non-capital and/or capital resources).

### 6.3.4. Reduction of overall risk due to transition to the alternative

[redacted]

### 6.3.5. Availability

The current LR2964 primer formulation failed to pass TRL 4, mainly due to insufficient corrosion performance during the Cyclic Heat / Salt spray and Neutral Salt Spray tests. Accordingly, identification of root cause failure will be carried out and afterwards re-formulation is necessary. As described in chapter 5.3.2, the development of this primer formulation to TRL9 will required a review period of significantly **more than 12 years**.

### 6.3.6. Conclusion on suitability and availability for Alternative 3

Due to the fact that technical performance in the most important and critical key functionality corrosion resistance is not given, the Cr(VI)-free primer formulation cannot be regarded a suitable alternative for Rockhard Chromate primer containing pentazinc chromate octahydroxide in safety critical aerospace and aeroderivative applications. The technical performance criteria for passing TRL4 were not met by the alternative (see Figure 17).

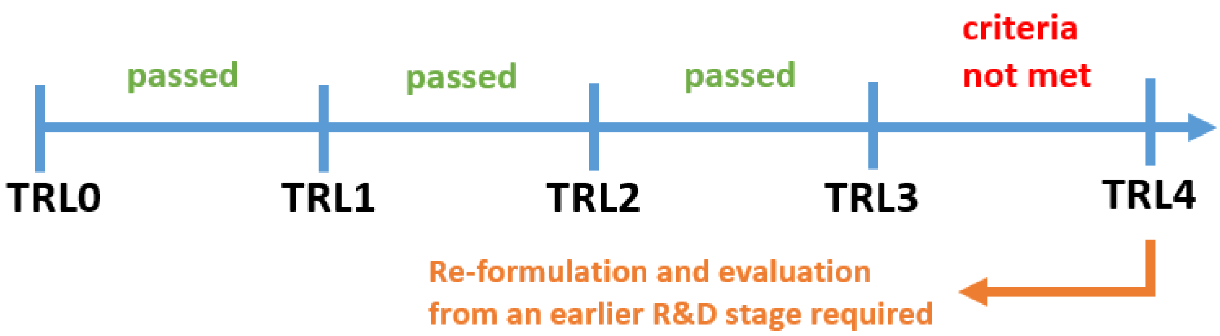


**Figure 17: Current R&D stage of LR2964 primer**

## 7. OVERALL CONCLUSIONS ON SUITABILITY AND AVAILABILITY OF POSSIBLE ALTERNATIVES FOR USE 1

Pentazinc chromate octahydroxide is used as corrosion inhibitor in the formulation of the Rockhard Chromate primer product. The product in scope is applied for production and MRO of aircraft engine components such as compressor casings, fan cases, oil tanks, gearboxes or couplings. Its corrosion inhibiting properties are extremely effective at low loadings (i.e. concentrations).Generally speaking, the purpose of these applications is to provide enhanced corrosion protection on such aircraft engine parts.

Significant research has been conducted within the aviation sector to identify and develop alternatives for Cr(VI) based applications. Alternatives for pentazinc chromate octahydroxide in primer formulations were assessed in the applicants' own research, R&D cooperation projects, intensive literature research and a consultation phase in which the information was compiled and evaluated for the AoA.

As result of the extensive R&D activities carried out by the applicant, 3 candidate alternatives were identified. The candidates are currently tested for TRL4. The technical alternative assessment was based on several key parameters which is summarises in Table 11 below.

**Table 11: Summary of the assessment of the alternatives for pentazinc chromate octahydroxide in primer applications for aircraft engine components**

| **Potential alternative** | **Assessment parameter** | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | **Corrosion resistance** | **Adhesion** | **Layer thickness** | **Chemical resistance** | **Temperature resistance** | **Compatibility (substrate / coating)** | **Flexibility** | **Scratch resistance** |
| LR2992 | | | | | | | | |
| LR2965 | | | | | | | | |
| LR2964 | | | | | | | | |

The corrosion inhibitors tested did, as far as the information is available, still show limitations in key requirements such as corrosion resistance and active corrosion inhibition. To successfully substitute pentazinc chromate octahydroxide for aircraft engine components, re-formulation and further testing of the candidates will be necessary before they successfully pass TRL4. Indeed, further research is planned or currently being conducted for each of them. Taking into account the development and approval process in the aviation sector, as described in Annex A together with the substitution efforts carried out so far by the applicants (see chapter 5.3.2), at least 14 years are required until an alternative for pentazinc chromate octahydroxide for applications in scope of this AoA is industrialised.

For all the reasons stated and with reference to the findings of the SEA, a review period of at least 12 years is considered to be adequate for the continued use of pentazinc chromate octahydroxide in the aviation industry.

## 8. REFERENCES

AMMTIAC (2012): Analysis of Alternatives to Hexavalent Chromium: A Program Management Guide to Minimize the Use of Cr(VI) in Military Systems.Richard A. Lane, Christopher Fink, and Christian Grethlein, P.E. Advanced Materials, Manufacturing, and Testing Information Analysis Center.

Federal Aviation Administration (FAA) (2012): Aviation Maintenance Technician Handbook—Airframe (FAA-H-8083-31).

ECHA and EASA (2014) An elaboration of key aspects of the authorisation process in the context of aviation industry. http://echa.europa.eu/documents/10162/13552/aviation_authorisation_final_en.pdf

US Department of Defence: Manufacturing Readiness Level (MRL) Deskbook, Version 2.0 May 2011; prepared by the OSD Manufacturing Technology Program in collaboration with the Joint Service / Industry MRL Working Group.

# ANNEX A – GENERAL OVERVIEW ON THE THE DEVELOPMENT AND APPROVAL PROCESS IN THE AEROSPACE INDUSTRY

## 8.1. General overview

Reliable and safe operation of complex aerospace systems depends upon the proper functioning of a multitude of components and materials. Failure of even a single small component can lead to undesirable events. For this reason, all changes to the materials, components, or manufacturing processes used in complex aerospace systems are subject to the highest level of scrutiny. No change is so minor that it does not require justification. Qualification, validation and certification of systems are applicable to a single specific configuration of components and materials, assembled by a single set of manufacturing processes. Any change to the components, materials, or manufacturing processes invalidates the qualification and certification.

Formal systems are in place to manage change, whereby all of the impacts of the change are analysed, and justifications must be provided for why the qualifications and certifications are still valid. Justification can take many forms. Trivial changes (like changing the characters used in marking) can be justified by simple analysis. Substantive changes, like those involving new surface finishes, require more substantial sets of data for justification. This almost always involves many levels of testing. It is therefore important to understand that even in the situation where a perfect alternative exists for specific design parameters, obtaining and documenting the data necessary to justify its implementation is a time-consuming and expensive process.

Much has already been written about the airworthiness and approvals process in the aerospace industry in the document “An elaboration of key aspects of the authorisation process in the context of aviation industry“ published in April 2014 by ECHA and European Aviation Safety Agency (EASA). The document makes a strong case for justification of long review periods for the aerospace industry. In this chapter we identify key points from the ECHA EASA “elaboration” document and add additional detail and justification for the required review period of 12 years for the continued use of pentazinc chromate octahydroxide in Rockhard Chromate primer for surface treatments of aircraft engine components for aerospace and aeroderivative applications.

Some of the key points identified in the “elaboration” document are:

- “The commercial aerospace industry must comply with the airworthiness requirements derived from EU Regulation No 216/2008 in Europe, and with similar airworthiness requirements in all countries where aeronautical products are sold.”

- “All components, from seats and galleys to bolts, equipment, materials and processes incorporated in an aircraft fulfil specific functions and must be certified, qualified and industrialised.” In addition, the new materials must be developed and evaluated prior to these three steps.
- “If a substance used in a material, process, component, or equipment, needs to be changed, this extensive process [of development, qualification, validation, certification and industrialization] has to be followed in order to be compliant with the airworthiness requirements.”
- “Although the airworthiness regulations (and associated Certification Specifications) do not specify materials or substances to be used, they set performance specifications [requirements] to be met (e.g. fire testing protocols, loads to be sustained, damage tolerance, corrosion control, etc.). These performance specifications [requirements] will drive the choice of substances to be used either directly in the aircraft or during the manufacturing and maintenance activities.”
- The development (Technology Readiness Level (TRL) 1-6) process “is an extensive internal approval process with many different steps from basic technology research up to technology demonstration in a lab environment.”
- “Depending upon the difficulty of the technical requirements [qualification] can easily take 3-5 years. After initial laboratory testing, each specific application must be reviewed, which means additional testing for specific applications / parts. Airworthiness Certification begins at this same time, this certification can take from 6 months to years. Additional time is needed for production scale-up and development of a supply chain.”

Each one of these points is of significant importance for the aerospace industry with regards to pentazinc chromate octahydroxide. Further elaboration will be made within this section.

In this chapter, the general process for alternative development through qualification, validation, certification, industrialisation and implementation within the aerospace industry is described. This process is also followed closely by the defence industries.

Defence hardware and systems are also subject to rigorous qualification requirements. Once a configuration has been qualified to the requirements of the controlling Ministry of Defence, changes cannot be made to the design or the manufacturing processes without requalification. For trivial changes, requalification may be obtained without the need to resort to testing. Significant changes, like those that would affect the corrosion performance of the system, typically require some amount of testing and analysis to provide requalification. Qualification and requalification of defence systems involves verification of performance to unique design parameters such as: resistance to chemical agent decontamination fluids, resistance to rocket motor exhaust, etc. Therefore, qualification and certification of a specific piece of hardware for civil requirements does not necessarily guarantee qualification for a specific defence application. Because of the stringent requirements for qualification and certification, a formal process for technology readiness and manufacturing readiness is followed.

The process for qualification, certification, and industrialization as described in the ECHA EASA "elaboration" document is shown in Figure 18.

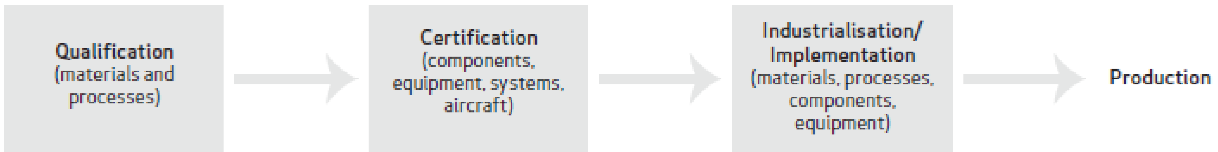


**Figure 18: Illustration of the qualification, certification and industrialisation processes.**

This diagram is perhaps overly simplified and doesn't indicate the significant level of R&D work required prior to achieving qualification. As stated in the "elaboration" document", this process is an extensive internal approval process with many different steps from basic technology research up to technology demonstration in a lab environment." The actual process followed by OEMs in the aerospace industry more closely follows the framework for TRLs and Manufacturing Readiness Levels (MRLs) originally developed by the National Aeronautics and Space Administration (NASA). OEMs usually adapt this TRL/MRL approach resulting in individual versions which are considered confidential and cannot be presented here. The NASA version is shown in Table 12.

Table 12: Technology Readiness Levels – Overview (US Department of Defence, 2011, adapted 2014).

| TRL# | Level Title | Description |
|---|---|---|
| 1 | Basic principles observed and reported | Lowest level of technology readiness. Scientific research begins to be translated into applied research and development (R&D). Examples might include paper studies of a technology's basic properties. |
| 2 | Technology concept and/or application formulated | Invention begins. Once basic principles are observed, practical applications can be invented. Applications are speculative, and there may be no proof or detailed analysis to support the assumptions. Examples are limited to analytic studies. |
| 3 | Analytical and experimental critical function and/or characteristic proof-of-concept | Active R&D is initiated. This includes analytical studies and laboratory studies to physically validate the analytical predictions of separate elements of the technology. Examples include components that are not yet integrated or representative. |
| 4 | Component and/or breadboard validation in laboratory environment | Basic technological components are integrated to establish that they will work together. This is relatively "low fidelity" |

| TRL# | Level Title | Description |
|---|---|---|
| | | compared with the eventual system. Examples include integration of “ad hoc” hardware in the laboratory. |
| 5 | Component and/or breadboard validation in relevant environment | Fidelity of breadboard technology increases significantly. The basic technological components are integrated with reasonably realistic supporting elements so they can be tested in a simulated environment. Examples include “high-fidelity” laboratory integration of components. |
| 6 | System / subsystem model or prototype demonstration in a relevant environment | Representative model or prototype system, which is well beyond that of TRL 5, is tested in a relevant environment. Represents a major step up in a technology’s demonstrated readiness. Examples include testing a prototype in a high-fidelity laboratory environment or in a simulated operational environment. |
| 7 | System prototype demonstration in an operational environment | Prototype near or at planned operational system. Represents a major step up from TRL 6 by requiring demonstration of an actual system prototype in an operational environment (e.g., in an aircraft, in a vehicle, or in space). |
| 8 | Actual system completed and qualified through test and demonstration | Technology has been proven to work in its final form and under expected conditions. In almost all cases, this TRL represents the end of true system development. Examples include developmental test and evaluation (DT&E) of the system in its intended weapon system to determine if it meets design specifications [requirements]. |
| 9 | Actual system through successful mission operations | Actual application of the technology in its final form and under mission conditions, such as those encountered in operational test and evaluation (OT&E). Examples include using the system under operational mission conditions. |

The TRL assessments guide engineers and management in deciding when a candidate alternative (be it a material or process) is ready to advance to the next level. Early in the process, technical experts establish basic criteria and deliverables required to proceed from one level to the next. As the technology matures, additional stakeholders become involved and the criteria are refined based on the relevant design parameters. Many more factors have to be taken into account prior to making a decision about transition of technology or replacing a material. A formal gate review process has been established by some companies to control passage between certain levels in the process.

Similarly, the maturity of manufacturing processes are formally tracked using the MRL process. Many companies combine the aspects of TRLs and MRLs in their maturity assessment criteria, as issues in either the technology or manufacturing development will determine production readiness and implementation of any new technology.

Figure 19 provides an overview of these key phases of introducing a candidate alternative into production hardware along with average timeframes based on aerospace experience.

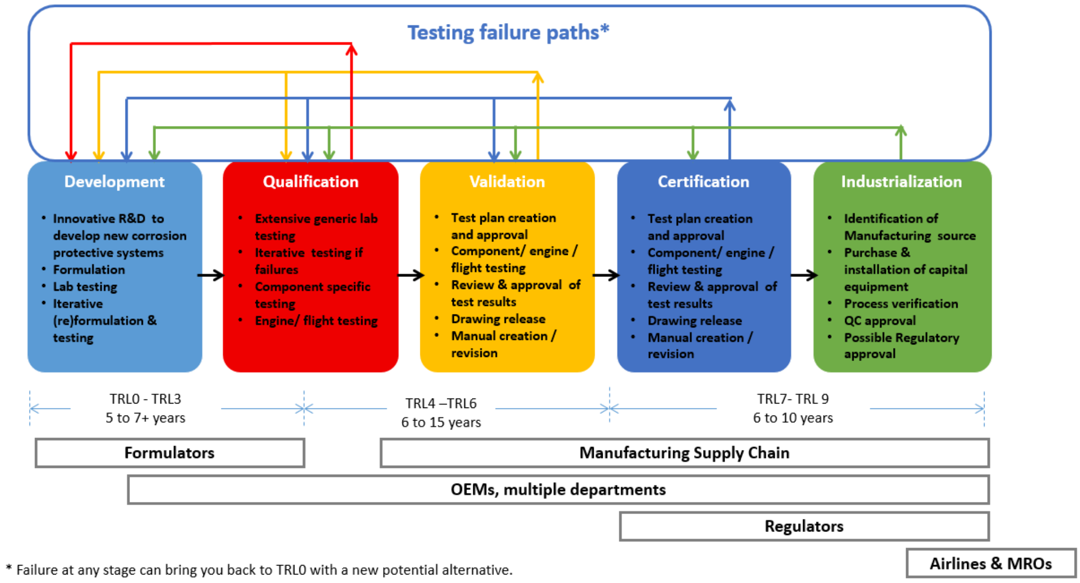


**Figure 19: Key phases of introducing a potential Cr(VI)-free alternative into production hardware.**

There is no guarantee that the initial process to identify an alternative for a substance will be successful. Failure is possible at every stage of the TRL and MRL processes. The impact of failure can be significant in terms of time.

The following sections describe the highlights of the entire process from definition of needs before technology development begins through to implementation.

## 8.2. Development, qualification and validation

The need for a design change may be triggered due to many reasons. The one of interest here is when a substance currently used for production of aerospace components is targeted for sunset (e.g. pentazinc chromate octahydroxide). Completely removing one substance may impact various parts and systems and may involve many different processes with different performance requirements.

Once a substance is identified to be targeted by a regulation, a first step is to identify the dependence upon the substance across the life cycle of products, including the materials and processes containing the specific substance in house, as well as within the supply chain and MRO activities. Most companies rely upon the information provided by the chemical manufacturer in the safety data sheet (SDS). This information source has many limitations when used for substance identification including: lack of reporting due to protection of confidential data; reporting large concentration bands to protect specific formulary data; different disclosure requirements based upon country (e.g., articles exemption, regulatory thresholds and de minimis cut-offs, specific substance classifications) to name a few. After identifying the relevant materials and processes and associating those with specifications and other design references, the affected hardware and related systems are identified. This is the first step in order to assess the impact for the company.

This work requires contributions from numerous personnel from various departments of an aerospace company including Materials & Processes, Research & Development, Engineering, Customer Service, Procurement, Manufacturing, Certification, as well as affiliates in other countries.

Current production aerospace components may have been designed 20 to 30 years ago (or more) using design methods and tools that are not easily revisited, nor were they necessarily standardized between OEMs. Checking and changing the drawings implies updating, e.g. creating the drawings under the new formats and tools, which can involve a tremendous amount of design work.

Please note: When a new design is needed (e.g. to remove a substance), it may not be compatible with the existing one; this means that spare part designs of the original materials / configurations may need to be preserved in order to be able to produce spare parts using the original (baseline) configuration. This is an additional impact to be taken into account.

The development and qualification process illustrated in Figure 19 is complex, and several years are often necessary.

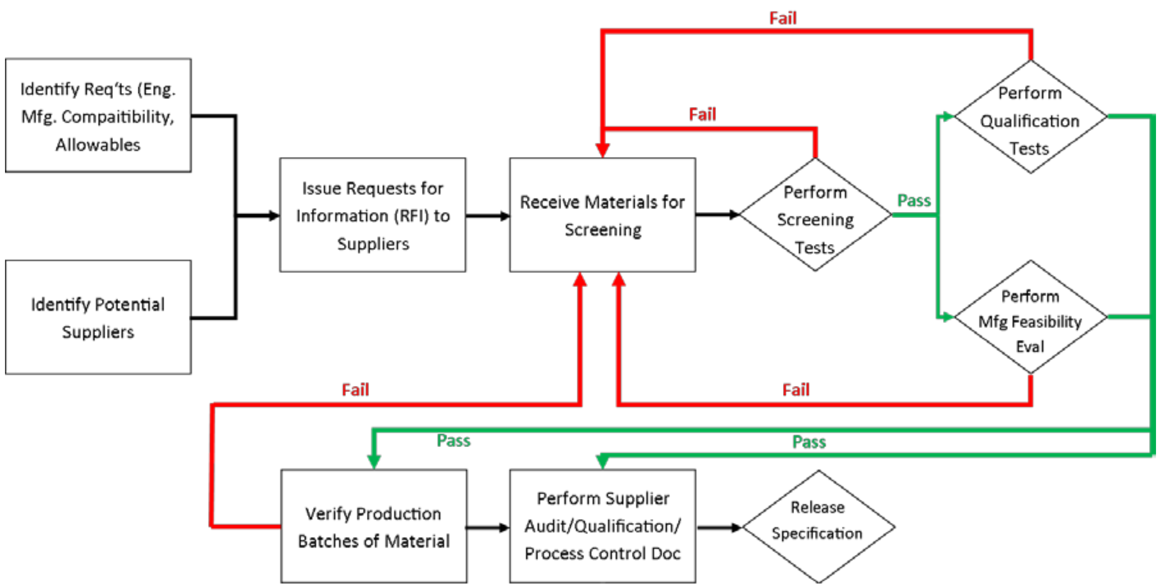


Figure 20: Illustration of the technology development and qualification process. (EASA, 2014)

### 8.2.1. Requirements development

Once a substitution project is launched, technical specialists, from engineering and manufacturing departments, must define the requirements that the alternatives have to fulfil.

Alternatives must satisfy numerous requirements. In many cases requirements are identified that introduce competing technical constraints and lead to complex test programmes. This can limit the evaluation of alternatives. For instance, for some materials, dozens of individual engineering requirements with similar quantities of industrial requirements may be defined.

Categories of technical requirements may include:

- Materials and processes requirements (e.g. corrosion resistance, adhesion strength)
- Design requirements (e.g. compatibility of the component's geometry complexity with the coating technique)
- Industrial requirements (e.g. robustness and repeatability)
- Environment, Health & Safety (EHS) requirements

Definition of requirements itself can be complex and requires a significant timeframe. The complexity can be due to:

- Different behaviour of the substitute compared to original product: new requirements may be defined. In this case, sufficient operational feedback to technically understand the phenomenon and to reproduce it at laboratory scale is a must in order to be able to define acceptance criteria.
- Requirements may come from suppliers and have an impact on the design.
- EHS regulations evolution.

Once initial technical requirements are defined, potential solutions can then be identified and tested. The timeframe for initial requirements development can last up to 6 months. Note that requirements may be added and continue to be refined during the different levels of maturity.

### 8.2.2. Formulation development

When formulators have no "off the shelf" solutions, they have to develop new ones, taking into consideration the complex aerospace design parameters identified in the requirements development step discussed above.

It is important to note that many iterations of these formulas are rejected in the formulator's laboratory and do not proceed to OEM evaluation. Formulators estimate that it typically takes 2 to 5 years of testing potential formulations before a candidate is identified for submittal to OEMs.

### 8.2.3. Technology development

The qualification process (typically TRL 3-4) is complex, and several years are often necessary before reaching qualification phase end (TRL 4).

> "This process is an extensive internal approval process with many different steps from basic technology research up to technology demonstration in a lab environment. Depending upon the difficulty of the technical requirements, these initial steps can easily take 3-5 years. After initial laboratory testing, each specific [aerospace] application must be reviewed, which means additional testing for specific applications [design parameters] / parts. Airworthiness Certification begins at this same time, this certification can take from 6 months to years. Additional time is needed for production scale-up and development of a supply chain."

The following points explain why it may be long and complex:

- Developing solutions usually necessitates several testing phases before meeting the numerous requirements, which often require several iterations to optimise the formulation / design.
- Some tests are long lasting (e.g. some corrosion tests last 3000 h or longer).
- In some cases, candidate alternatives are patented, preventing multiple sources of supply, which is an obstacle to a large supply-chain deployment.
- When no broad replacement is available, each alternative must be evaluated case-by-case to determine which, if any, are suitable for specific design parameters.
- Because there is no single alternative for Cr(VI), the industry is forced to replace one formulation with multiple alternatives. This exacerbates the already complicated configuration management of complex aerospace systems.
    - Multiple different drawing requirements need to be managed in the design.
    - Multiple different manufacturing and maintenance processes need to be certified and controlled.
- Substance-specific regulations are evolving throughout the long R&D phase and life cycle of aerospace components, which could result in an unintended regrettable substitution (e.g., boric acid as a substitute for chromic acid in anodising).
- The replacement of a material / process may impact the complete design of a part. Additionally, the mating part / counterpart functionality must be analysed (materials compatibility, dimensional compatibility, stress compatibility). This may lead to redesign of the complete part plus mating parts.

– Even after 30 plus years of significant investment in R&D, it is apparent that time and money cannot guarantee successful innovation.

As stated in the “elaboration” document:

> “Once candidate(s) are developed, the OEM evaluates candidates by performing screening testing. If the candidate passes screening, testing is expanded to increase the likelihood that the preparation will pass qualification. If the candidate fails, which is often the case, material suppliers may choose to reformulate. It is not uncommon to iterate multiple times before a candidate passes screening. In some technically challenging areas, over 100 formulations have been tested with no success. This phase of development can take multiple years depending upon the material requirements.”

It should be noted that the timeframes for development and qualification stated in the “elaboration” document have been combined and may be understated in the case of pentazinc chromate octahydroxide. Depending on the design parameters and the complexity of material and process requirements, this process can easily take multiple years.  As noted in the “elaboration” document, the timeframe for development alone is typically a minimum of 3 to 5 years. Our experience with replacement of the substance addressed in this dossier is that the development takes much longer. Typically for successful projects, the duration is 3 to 5 years. For unsuccessful projects, the development goes through repeated iterations and has taken over 30 years and still continues with limited success.

### 8.2.4. Qualification and Validation

All materials, components, equipment or processes have to meet or exceed the specific performance requirements which are defined in the Certification Specifications documented in technical standards as described in chapter 3.3.3. These are issued by defence organisations, government-accredited bodies, industry associations or based upon company-developed confidential specifications.

The main reasons for qualification are:

– To fulfil requirements by the Airworthiness Authorities (EASA); this is the first level of the Aircraft Certification Pyramid.

– To ensure that only approved, reliably performing materials, parts and processes are used to produce aerospace components.

– To ensure that the product, the process or method is compliant with the Industry Regulations and aerospace components manufacturer requirements to fulfil a specified function.

- To provide a level of confidence and safety.
- To ensure consistent quality of products and processes.
- To ensure supplier control, and to guarantee production and management system robustness, throughout the supply chain.

The qualification process is mandatory to demonstrate compliance with airworthiness and certification requirements; the qualification process ensures that the technical and manufacturing requirements documented in the relevant material and/or process specifications are met. The qualification process comprises several steps before materials / processes are qualified. Even if most showstoppers are identified during the development phase, process confirmation / production verification are performed during the qualification phase. In case of failure, product qualification will be cancelled and the development phase must start again from the beginning.

> "For those materials that pass screening, production scale-up, development of process control documents, manufacturing site qualifications, and extensive qualification testing is required to demonstrate equivalent or better performance to that which is being replaced. This phase of the process can also result in formulation or manufacturing iterations and may take several additional years. Depending on the complexity of the change and the criticality of the application [design parameters] (for example, fire protection or corrosion prevention have high safety implications and require development and testing against multiple, rigorous performance standards), re-certification may be required."

Once an alternative is qualified, the performance of each particular aerospace application is validated based on its specific design criteria.

Based upon OEM experience, the time period needed to pass qualification and validation is estimated to be on the order of 6 to 15 years and can be even longer when major test failures occur. This is one of the main challenges for pentazinc chromate octahydroxide replacement.

### 8.3. Certification

This next step is to certify that aerospace components comply with all applicable airworthiness regulations and associated Certification Specifications (specs). This step is described in the "elaboration" document and is reproduced here for continuity.

> "Certification is the process under which it is determined that an aircraft, engine, propeller or any other aircraft part or equipment comply with the safety, performance environmental (noise

& emissions) and any other requirements contained in the applicable airworthiness regulations, like flammability, corrosion resistance etc.

Although the airworthiness regulations (and associated Certification Specifications) do not specify materials or substances to be used, they set performance specifications to be met (e.g. fire testing protocols, loads to be sustained, damage tolerance, corrosion control, etc.). These performance specifications will drive the choice of substances to be used either directly in the aircraft or during the manufacturing and maintenance activities. Some examples of performance requirements are the following:

- Resistance to deterioration (e.g. corrosion)
    - Environmental damage (corrosion for metal, delamination for composites) and accidental damage during operation or maintenance.
    - Corrosive fluids - Hydraulic fluids; Blue water systems (toilet systems and areas); leakage of corrosive fluids/substances from cargo.
    - Microbiological growth in aircraft fuel tanks due to moisture/contamination in fuel cause severe corrosion. Such corrosion debris has the potential to dislodge from the fuel tanks, migrate through the fuel system, and lead to an in-flight engine shutdown.
- Resistance to fire – Flammability Requirements
    - Fire-proof and fire-resistance. Aircraft elements are expected to withstand fire for a specified time without producing toxic fumes; this leads to using products like flame retardants, insulation blankets, heat protection elements in hot areas (e.g. around engines).

The primary certification of the aircraft (or engine and propeller) is granted to the manufacturer by the Competent Aviation Authority of the “State of Design” which is typically the authority of the state where the manufacturer of the aircraft (or engine or propeller) is officially located (EASA in the case of aircraft designed and manufactured in the EU and European Free Trade Association countries). Aircraft that are exported to other countries will have to be certified (validated) also by the authority of the “State of Registry”.

Manufacturers work with the certification authorities to develop a comprehensive plan to demonstrate that the aircraft meets the airworthiness requirements. This activity begins during the initial design phase and addresses the aircraft structure and all systems in normal and specific failure conditions (e.g. tire failure, failure of structural components, hydraulics, electrical or engines). The tests needed to demonstrate compliance, range from thousands of coupon tests of materials, parts and components of the airplane, up to tests that include the complete aircraft or represents the complete aircraft (system). The performance and durability of the various materials have to be confirmed while the behaviour of the parts, components and the complete airplane will have to be tested in the applicable environmental and flight conditions including various potential damage or failure conditions. For a new Type Certificate, this overall compliance demonstration covers several thousands of individual test plans of which some will require several years to complete. Often, after the initial issuance of the Type Certificate, the tests that have the objective to demonstrate durability of the aircraft during its service life will continue.

All the different aspects covered by the Type Certificate together define the “approved type design” which includes, among other aspects, all the materials and processes used during manufacturing and maintenance activities. Each individual aircraft has to be produced and maintained in conformity with this approved type design.

Changes to the approved type design may be driven by product improvements, improved manufacturing processes, new regulations (including those such as new authorisation requirements under REACH), customer options or the need to perform certain repairs. When new materials or design changes are introduced, the original compliance demonstration will have to be reviewed for applicability and validity, in addition to a review of potential new aspects of the new material or design change that could affect the airworthiness of the aircraft. Depending on the change, this review could be restricted to coupon or component tests, but for other changes this could involve rather extensive testing. E.g. changes in protective coatings could affect not only the corrosion resistance but could also affect the friction characteristics of moving components in actuators in the different environmental conditions, changing the dynamic behaviour of the system, which in the end affects the dynamic response of the airplane.

Before the new material or design change can be introduced on the aircraft, all test and compliance demonstrations have to be successfully completed and approved by the Competent Authority. This approval results in the issuance of a Supplemental Type Certificate (STC), change approval or repair approval.

> It is important to note that, according to the EU Regulation No 216/2008, EASA is the design competent authority for civil aircraft only. Any other aircraft (e.g. military, fire-fighting, state and police aircraft) will have to follow similar rules of the corresponding State of Registry.
>
> To be able to maintain and operate an aircraft the responsible organisations must be approved by the competent authority and compliance is verified on a regular basis. Maintenance of an aircraft requires that the organization complies with specific procedures and materials described in the maintenance manuals which are issued by and the responsibility of the OEMs."

Importantly, even if an alternative is in use in one component in aerospace system A, it cannot be inserted into what appears to be the same part in another aerospace system B (e.g., model B) until it is fully reviewed / validated / certified to ensure that either the design parameters are identical or that the alternative is fully acceptable for the different design parameters. Extensive experience shows that an alternative that is successfully certified in one component in one model cannot necessarily be successfully certified in another. In other words, the circumstances for each component in each model are unique and extrapolation is impossible without validation and certification.

As noted in the "elaboration" document, in optimal cases certification can take as little as 6 months but typically will take several years. The duration really depends on the specific hardware and design parameters.

It is important to understand that the Type Certificate is issued for the original design of the product in civil aviation (airframe, engine or propeller) as a whole, rather than for each part. However, every component part of the product must be designed, developed, and validated to meet the requirements of the overall product requirement and system design (how each component fits and interacts with other component parts). This approval is granted after the airworthiness certification criteria, compliance standards / requirements and methods of compliance have been successfully demonstrated to the relevant Airworthiness Authority.

Any change to the type certified product design must be evaluated and approved by the Type Certificate holder and Airworthiness Authority on the same basis to assure overall safety for the product to demonstrate overall airworthiness once integrated into the overall product design. If determined to be equivalent or better, the new configuration is certified.

The above responsibilities and obligations are defined in EU regulation 748/2012 for Type Certificate holders in the EU. When the state of design (geographic location of the Type Certificate) is in the

United States, the responsibility and obligations are defined in the U.S Federal Aviation Regulations (FARs).

Standard parts, such as a nut or bolt, must be manufactured in compliance with a government, established industry, or company standard. For many standard parts, specific manufacturers have been qualified as approved sources. Once qualified, no modifications to basic methods of manufacture, plant site, or quality level can be made without prior notification and approval from the OEM.

There are industry standards and specifications for materials, processes and standard parts; however, in many cases, the requirements are built upon consensus negotiated in an industry-wide committee. As a practical means of enabling manufacturing process quality control and acceptance testing of production batches, corrosion resistance specifications typically require a short duration test that is capable of identifying common defects in processing but are not suitable for initial alternative validation acceptance criteria. In order to reach consensus, the requirements may be less stringent than those required by individual companies. In such cases, an individual company will modify an industry standard, creating company-confidential specifications with more stringent and specific requirements to meet their product needs and regulatory requirements. These company specifications are confidential due to the investment of significant resources and intellectual property required to develop materials and processes to meet these more stringent requirements. Qualifications required to meet these confidential specifications are company specific. In very few cases, are the industry standards sufficient to meet all OEM requirements, thus the reliance upon company specifications.

### 8.4. Implementation / industrialisation

Aerospace company products consist of thousands to several million parts provided by thousands of suppliers or manufactured internally by OEMs. Significant investment, worker training and manufacturing documentation may be required to adapt the manufacturing processes, which sometimes require changes in existing facilities or the construction of new facilities.

The industrial implementation is usually scheduled to follow a stepwise approach to minimize the technical risks and benefit from lessons learned. This implies that the replacement is not implemented in one shot in all plants and at all suppliers but stepwise. Each OEM may operate dozens of manufacturing sites / final assembly lines worldwide.

Furthermore, the implementation of an alternative process may induce new development and modification in the complete process flow.

For legacy parts, long-term agreements are often in place with suppliers. When a change is made to a drawing to incorporate a new alternative, the contract with the supplier needs to be renegotiated, and additional costs are incurred by the supplier when modifying a production process and/or introducing a new process. These may include purchase and installation of new equipment, training of staff, internal qualification of the new process, OEM qualification of the supplier, manufacturing certification of the supplier, etc. In addition, the supplier may need to retain the ability to use the old coating system for other customers / hardware, which requires the supplier maintain parallel lines to accommodate both coatings / processes. The level of complexity varies by type of part / part family, process, etc.

The following text is reproduced from the "elaboration" document and describes the process for implementation of an alternative:

> "Industrialisation is an extensive step-by-step methodology followed in order to implement a qualified material or process throughout the manufacturing, supply chain and maintenance operations, leading to the final certification of the aerospace product. This includes re-negotiation with suppliers, investment in process implementation and final audit in order to qualify the processor to the qualified process.
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> Taking into account that an aircraft is assembled from several million parts provided by several thousand suppliers, this provides an indication of the complexity for the industrialisation stage of replacement materials / processes, and the supply chain which provides these parts.
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> Special challenges are:
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> - Low volumes limit influence on changes to suppliers' materials / processes;
> - Procurement & insertion of new equipment;
> - Scale-up & certification of new process;
> - Incompatibility of coatings could be a risk;
> - Re-negotiation of long term agreements with suppliers[2];
> - Increased complexity of repairs – Multiple solutions for different coatings / surface treatments as a substitute for a single, robust process. For example, currently all

[2] Changes to the design or manufacturing may require re-negotiations with suppliers, which can be time-consuming, especially when long-term contracts are concerned. The supply chain is complex in the aerospace industry; it includes but is not limited to chemical manufacturers, importers, distributors, formulators, component manufacturers, OEMs, Airline operators, and aftermarket repair and overhaul activities.

aluminium parts can be repaired with one chromated conversion coating. In some specific cases, the future state could require different conversion coatings for each aluminium alloy and unique design parameters. Since different aluminium alloys are not easily distinguishable on the shop floor, ensuring that the proper repair procedures are used will be much more difficult. If alternate means of compliance approvals are requested for repair facilities or airlines, regulatory agencies are unlikely to have adequate knowledge or technical data to make informed assessments.

The operating environment, longevity of the aircraft, supply chain complexity, performance and above all airworthiness requirements are some of the considerations which can constrain the ability of the industry to make changes and adopt substitutes in the short, medium or long term."

The timeframe for implementation and industrialisation is unknown. Even simple changes can take up to 5 years. When more than one alternative process is introduced simultaneously, up to a decade or more may be necessary to fully implement the alternatives.

It is important to note that the implementation / industrialisation step ('TRL10') refers to the whole supply chain. This includes external as well as internal industrialisation. In case a suitable Cr(VI)-free alternative is developed in the future, it needs to be implemented across a vast and complicated supply chain, which in turn is time and cost intensive requiring significant additional investment in new machinery and plants on the part of existing suppliers. Additionally, any substitution may be linked to major resourcing efforts to identify new suppliers with the capabilities of industrialising the new processes. This transition requires validating and qualifying the new alternative process at the new supplier(s).

### 8.5. Implementation at customers and maintenance, repair and operations (MROs)

Implementation by customers (e.g., airlines, navies, etc.) and Maintenance, Repair and Operations (MROs) further requires that an alternative be approved and certified and made available in the maintenance documents. This includes both scheduled and unscheduled MRO activities. Prior to including a validated alternative in the maintenance documents, similar steps as those described above need to be undertaken. MRO activities on in-service hardware is more complex due to restricted access to hardware as compared to access during manufacture and assembly. As such, all of these conditions must be addressed in the MRO certification process. Furthermore, once a part is in-service, additional unplanned repairs may be required and subsequently qualified, validated and certified before implementing. However, the effort for detailed development and testing for each individual component and its design parameters is necessary.

When the alternative process is included in the maintenance documents, customers and MROs face similar challenges to implement the alternative. Here, for operating supplies and testing timeframes, another 3 years might be necessary, depending on the complexity of the alternative.

In the aerospace industry, the methods for MRO are evaluated and determined by the OEMs.