Chemical Data Sheet Nº CDS-16

Title: Preparation & Control of AEP 32 & AEP 100 Process Baths

Related Procedure: VAIL-OPS-039

Revision: 1 **Date:** 24th June 2022

1.0 Health, Safety & Environmental Considerations

Prior to handling the various constituent solvents, powders, chemicals and additives used to make up the AEP process baths personnel must be familiar with the relevant MSDS and any COSHH assessments for these chemicals, refer to the 'sevron' database. The necessary precautions must be taken.

2.0 Product Specifications

AEP 32 & AEP 100 Process Baths

CDS-16-1 AEP 32 Process Bath Specifications
CDS-16-2 AEP 100 Process Bath Specifications

See Table 1

TABLE 1

AEP Process Bath Specifications and Green Coat Details

Designation:	**CDS-16-1**		**CDS-16-2**	
Process bath description:	**AEP 32**		**AEP 100**	
Rolls-Royce specification: [1]	**EPS 10040**		**EPS 10654**	
AEP base solvent				
Isopropanol weight %	60 ± 5 %		60 ± 5 %	
Nitromethane weight %	40 ± 5 %		40 ± 5 %	
Isopropanol volume %	68.5 ± 4.5 %		68.5 ± 4.5 %	
Nitromethane volume %	31.5 ± 4.5 %		31.5 ± 4.5 %	
AEP bath properties				
Specific gravity (SG)	0.880 - 0.915 g cm^{-3}		0.880 - 0.915 g cm^{-3}	
Operating temperature	16 – 32 °C		16 - 32 °C	
Water content	< 2 %		< 2 %	
AEP bath constituents				
	weight %	**conc / g/l**	**weight %**	**conc g/l**
zein (R-R spec EMS 56714) [2/3]		2.0 – 3.0 [2]		1.7 - 2.3 [2]
cobalt nitrate hexahydrate [2]		0.10 – 0.20 [2]		0.09 - 0.15 [2]
TOTAL pre-alloyed powder		15 – 50		14 - 50
INITIAL / OPTIMUM powder [4]		30 - 35		25 - 30
pre-alloyed powder details				
R-R specification	EMS 56729		EMS 56728	
powder type	Praxair AL-123		Praxair AL-131	
aluminium	38 – 46 [5]	6 – 22 [5]	68 - 72	10 – 35
chromium	36 – 44 [5]	6 – 21 [5]	28 - 32	4 – 16
manganese	14 – 22	3 – 10	*n/a*	*n/a*
AEP 'green coat' requirements				
aluminium	38 – 46 [5]		68 - 72	
chromium	36 – 44 [5]		28 - 32	
manganese	14 - 22		*n/a*	

NOTES:

[1] This process is also covered in some T56/501 engine overhaul manuals i.e.
TO 2J-T56-53 WP 207 00 / WP 208 00
TVL 2JA3-406-006-3 SECTION 10; Para 10.14
NAVAIR 02B-5DH-6-2V1 WP 009 00 Para 35.

[2] Ratio of cobalt nitrate hexahydrate (g/l) / zein (g/l) should be as close to 15 as possible.

[3] Zein – each new batch of zein powder must be qualified as outlined in paragraph 5.4.

[4] Initial make up and optimum operating pre-alloyed powder content.

[5] Weight percentage of chromium shall be less than or equal to weight percentage of aluminium in powder.

3.0 Requirements Summary

3.1. ***DAILY***
Local Operating Personnel
When processing check process bath level, pneumatic motor operation, pneumatic motor oil level (not required if using 'dry' motors), stirrer speed, coating room temperature, coating room humidity, rectifier operation and all safety features.

3.2. ***WEEKLY***
Chemical Analysis (Laboratory)
Check total alloy powder (metal), cobalt nitrate hexahydrate & zein content (g/l). Use gravimetric analysis methods for total alloy content and zein. Use AAS method for cobalt nitrate hexahydrate. Check solvent SG.

3.3. ***PERIODICALLY***
Chemical Analysis (Laboratory)
Verify individual concentrations and ratios of aluminium, chromium & manganese [6] and calculate total alloy powder (metal) content using the AAS method. [7]
NOTES:
[6] Mn, AEP 32 only
[7] Approximately every 4 – 6 weeks depending on bath utilization
Determine water content using an appropriate external approved laboratory.

3.4. ***IF REQUIRED***
Chemical Analysis (Laboratory)
Carry out 'green coat' analysis if required for troubleshooting purposes only, not required for process control.

3.5. AEP coating baths must be maintained in working condition with all constituents in balance and well dispersed in the media. These process baths should be replaced when they cease to operate effectively. Depending on usage and environmental conditions the process bath solutions should be replaced at least every six months to one year.

4.0 Process Tank Details

Coating Facilities & Process Tank Details

4.1. **The electrophoretic coating process uses a high DC voltage in the application of the coating. The coating facilities must be designed and constructed to provide the operation with maximum protection from electric shock.**

The electrophoretic coating bath is flammable and emits vapours which can reach an explosive concentration. Excessive breathing of the fumes may cause toxic effects. The exhaust blower must be on at all times while the coating room is occupied and must be on during all phases of the coating operation. The AEP process baths must be kept covered when not in use to minimise evaporation.

4.2. The coating room must be fitted with a suitable air conditioning system to maintain the following at all times :-

temperature:	**16 - 32 °C**
humidity:	**35 - 50 %**

4.3. All AC and DC electrical equipment installed in the coating room must conform to latest electrical & insulation regulations to avoid any sparks or fire hazards.

4.4. The coating room must be fitted with a fully serviceable automatic discharge carbon dioxide fire extinguishing system which must be capable of being disabled if any persons are inside the room.

4.5. The AEP coating tanks shall be stainless steel with continuously running pneumatically operated agitation; ~ 40 – 45 rpm to maintain the fine powder in suspension. The tanks must have lids to prevent evaporation and water pick up.

4.6. The AEP coating facility must be fitted with suitably compliant rectifiers to generate the required DC current.

5.0 Product Preparation

AEP Process Bath Preparation

5.1. AEP process baths are prepared in several steps, read complete sections, paragraphs 4.0 to 6.0, before commencing bath preparation to ensure that all the necessary materials are available and suitably qualified. Carry out ***STEPS 1 to 3*** prior to making up AEP process baths.

5.2. Materials required

Isopropanol 99 % min (anhydrous)		200 l drum
Nitromethane 95 % min		200 l drum
Zein (corn gluten) – low electrolyte grade	EMS 56714	1 kg pack *typical, pack size varies*
Cobalt nitrate hexahydrate (reagent grade)		250 g (typical)
Praxair AL 123 pre-alloyed powder (Al/Cr/Mn)	EMS 56729	10 lb (4.54 kg) pack
Praxair AL 131 pre-alloyed powder (Al/Cr)	EMS 56728	10 lb (4.54 kg) pack

5.3. Preparation of AEP Process Baths

See also Table 2, AEP Process Bath Preparation Summary

5.3.1. ***STEP 1*** - Base solvent, isopropanol/nitromethane,

These solvents are flammable, therefore care must be exercised to prevent ignition before, during & after mixing. To prevent plasticizer contamination, avoid the use of plastics such as PVC or Tygon.

Bulk mix the base solvent in a clean tank (may use AEP coating tank) in the following volume ratios :-

Isopropanol	**70 %**	(equivalent weight %, **61.6**)
Nitromethane	**30 %**	(equivalent weight %, **38.4**)

e.g.

AEP 32 130 litre Tank - Mix **110 litres; 77 l** isopropanol & **33 l** nitromethane
AEP 100 130 litre Tank - Mix **114 litres; 80 l** isopropanol & **34 l** nitromethane
Using a suitable hydrometer check that SG is **0.880 – 0.915 g cm^{-3}**
If SG is high (> 0.915 g cm^{-3}) add more isopropanol (SG 0.785 g cm^{-3})
If SG is low (< 0.880 g cm^{-3}) add more nitromethane (SG 1.140 g cm^{-3})

5.3.1. *(continued)*

Always keep solvents covered to avoid evaporation and water pick up.
Make up more base solvent as required for further additions and top-ups in the above ratio and store in 200 litre steel drums &/or 25 litre plastic drums. Ensure all drums are sealed and correctly labelled with contents and mixing date.

5.3.2. ***STEP 2*** – Zein solution

Using zein preparation tank, mix **35 litres** of isopropanol & **15 litres** of nitromethane to produce **50 litres** of base solvent as above or take **50 litres** of base solvent prepared above. Slowly dissolve approx **850 g** of qualified zein [8] while using sufficient agitation to dissolve the zein but not to aerate the solution. This should give a zein concentration of about **17 g/l**. Allow this solution to stand for 24 hours.

NOTE: *[8] Every new batch of zein powder must be qualified before use, see paragraph 5.4 below.*

Filter zein solution using Whatman 113V filter papers (or equivalent) into clean containers. The containers must be capped to avoid evaporation and water pick up. Recommended to use 25 l plastic drums.

Determine the zein concentration:-

- Pipette 3 X 10 ml samples from the zein solution (***STEP 2***) into pre-weighed aluminium dishes
- Evaporate to dryness (constant weight) – slowly to avoid burning
- Cool and weigh dish to determine weight of zein

*zein concentration **z** (g/l) = weight of zein (g) x **100***

Record the average zein concentration from the 3 samples.

Allocate a batch number to this zein solution e.g. "**19/09/17 – 1**" and clearly label containers with this batch number and the zein concentration **z** in g/l determined above. These containers may be stored and used for future AEP bath additions and make ups.

Also record details of this zein solution (batch, volume & zein concentration) and the details of the zein qualification (zein batch number & water solubility) on *AEP Process Baths Preparation Log Sheet, Part A (Form VAIL-OPS-039F02)*.

5.3.3. ***STEP 3*** - Cobalt nitrate hexahydrate solution

Decant **5 litres** of base solvent, ***STEP 1*** (do not use zein solution, ***STEP 2***) from AEP tank or holding drum into a suitable large glass beaker, dissolve **19 g** of cobalt nitrate hexahydrate into solution. This gives a solution of **3.8 g/l** cobalt nitrate hexahydrate.

5.3.4. Typical **AEP 32** tank make up (Fleetlands, Building 112, 130 litre tank)

Fill tank with approx **105 litres** [9] of base solvent (***STEP 1***) and start pneumatic stirrers at ~ 45 RPM.

NOTE: *[9]Initial 110 litre with 5 litres decanted for cobalt nitrate solution* (***STEP 3***).

Add sufficient amount of zein solution (***STEP 2***) to produce the required zein concentration.

weight of zein required (g) =
*tank volume **T** (l) x zein concentration required (g/l)*

*volume of zein solution (**STEP 2**) required (l) =*
*weight of zein required (g) / zein solution concentration **z** (g/l)*

*Add **19 litres** of **17 g/l** zein solution (**STEP 2**) to **AEP 32** tank.*

NOTE: *If zein solution is **17 g/l** zein **19 litres** gives **2.5 g/l** zein in a 130 litre AEP 32 bath*

Add sufficient amount of cobalt nitrate hexahydrate solution (***STEP 3***) to produce the required cobalt nitrate hexahydrate concentration.

*volume of cobalt nitrate hexahydrate solution (**STEP 3**) required (l) =*
*{tank volume **T** (l) x **0.15**} / **3.8***

*Add **5 litres** of **3.8 g/l** cobalt nitrate hexahydrate solution (**STEP 3**) to **AEP 32** tank*

NOTE: *If cobalt nitrate hexahydrate solution is **3.8 g/l** cobalt nitrate hexahydrate **5 litres** gives **0.146 g/l** cobalt nitrate hexahydrate in a 130 litre AEP 32 bath*

Very slowly add required amount of Praxair **AL-123** pre-alloyed powder to the coating tank.

*Praxair AL-123 powder required (g) = **34** x tank volume **T** (l)*

*Slowly add **4.54 kg Praxair AL-123** pre-alloyed powder to **AEP 32** tank*

NOTE: *This gives a total alloy powder content of **~ 34.9 g/l***

Finally, if required, add sufficient base solvent (***STEP 1***) from holding drum to bring AEP 32 process bath up to final level.

5.3.5. Typical **AEP 100** tank make up (Fleetlands, Building 112, 130 litre tank)

Fill tank with approx **109 litres** [10] of base solvent (***STEP 1***) and start pneumatic stirrers at ~ 45 RPM.

> ***NOTE:*** [10]*Initial 114 litre with 5 litres decanted for cobalt nitrate solution* (***STEP 3***).

Add sufficient amount of zein solution (***STEP 2***) to produce the required zein concentration.

weight of zein required (g) =
*tank volume **T** (l) x zein concentration required (g/l)*

*volume of zein solution (**STEP 2**) required (l) =*
*weight of zein required (g) / zein solution concentration **z** (g/l)*

*Add **16 litres** of **17 g/l** zein solution (**STEP 2**) to **AEP 100** tank.*

> ***NOTE:*** *If zein solution is **17 g/l** zein **16 litres** gives **2.1 g/l** zein in a 130 litre AEP 100 bath*

Add sufficient amount of cobalt nitrate solution (***STEP 3***) to produce the required cobalt nitrate hexahydrate concentration.

*volume of cobalt nitrate hexahydrate solution (**STEP 3**) required (l) =*
*{tank volume **T** (l) x **0.15**} / **3.8***

*Add **5 litres** of **3.8 g/l** cobalt nitrate hexahydrate solution (**STEP 3**) to **AEP 100** tank*

> ***NOTE:*** *If cobalt nitrate hexahydrate solution is **3.8 g/l** cobalt nitrate hexahydrate, **5 litres** gives **0.146 g/l** cobalt nitrate hexahydrate in a 130 litre AEP 100 bath*

Very slowly add required amount of Praxair **AL-131** pre-alloyed powder to the coating tank

*Praxair AL-131 powder required (g) = **23** x tank volume **T** (l)*

*Slowly add **3 kg Praxair AL-131** pre-alloyed powder to **AEP 100** tank*

> ***NOTE:*** *This gives a total alloy powder content of **23 g/l***

Finally, if required, add sufficient base solvent (***STEP 1***) from holding drum to bring AEP 100 process bath up to final level.

5.3.6. The AEP coating bath is now ready to use but should be left for 24 hours before AEP coating, the agitation must be left running continuously at about 40 – 45 rpm.

5.3.7. Carry out a full chemical analysis of the AEP bath as outlined in paragraph 7.0.

5.3.8. Record details of the above solution make up on Part B of *AEP Process Baths Preparation Log Sheet (*Form VAIL-OPS-039F02*)*.

5.3.9. Any remaining drums of base solvent (***STEP 1***) or containers of zein solution

(***STEP 2***) may be stored for future use. They must be sealed and clearly labelled.

TABLE 2

AEP Process Bath Preparation Summary

Constituent Additions		To make up 130 litres AEP 32 solution			To make up 130 litres AEP 100 solution		
		Vol / litres	**Weight**	**Conc / g/l**	**Vol / litres**	**Weight**	**Conc / g/l**
Isopropanol & nitromethane base solvent	***STEP 1***	**105**	*93.608 kg*	-	**109**	*97.174 kg*	-
Zein solution	***STEP 2***	**19**	*325 g zein*	*17 g/l (soln)* 2.5 g/l zein	**16**	*272 g zein*	*17 g/l (soln)* 2.1 g/l zein
Cobalt nitrate hexahydrate solution	***STEP 3***	**5**	*19 g cobalt nitrate hexahydrate*	*3.8 g/l (soln)* 0.146 g/l cobalt nitrate hexahydrate	**5**	*19 g cobalt nitrate hexahydrate*	*3.8 g/l (soln)* 0.146 g/l cobalt nitrate hexahydrate
Pre-alloyed powder	**Praxair AL-123**	-	**4.54 kg**	34.9 g/l AL-123			
	Praxair AL-131				-	**3.00 kg**	23.0 g/l AL-131
Base solvent top up (if req'd)	***STEP 1***	**1**	*891.5 g*	-	**-**	-	-

NOTE: *The above table summarises the constituents of each bath, prior to making up baths read and understand paragraphs 4.0 – 6.0, Ensure ALL of **STEPS 1**, **2** & **3** are completed prior to making up process baths.*

5.4. Zein Qualification

5.4.1. The following test shall be carried out on each new batch of zein received. If the zein does not reach the criteria below then it is NOT acceptable for use in AEP solutions. Water solubility shall not exceed 4%.

- Take 3 g of zein, wash through filter paper into a beaker with 100 ml DI water
- Wash a second time with 100 ml DI water

NOTE: *Water filtrate shall be essentially colourless, a slight cloudiness is acceptable*

- Pipette 25 ml into a weighed dish, evaporate to dryness and reweigh dish
- Calculate % water solubility of zein :-

$$\mathbf{100} - (\mathbf{0.375} / wt \times \mathbf{100})$$

- Record zein batch number and water solubility as outlined in ***STEP 2*** above.

5.4.2. Current density test

- Using a 2 litre test beaker formulate the following test sample

40 g	Praxair AL 123 Al/Cr/Mn powder
4.4 g	test zein
0.28 g	cobalt nitrate hexahydrate
balance	70:30 (volume) isopropanol : nitromethane solvent

- Using a Rolls-Royce T56/501 2nd stage turbine blade (surface area 31 cm^2) and the power source, set a voltage to achieve a current of **0.062 Amps**.

- The current density shall drop to half its initial value in 3 minutes or less

6.0 Product Maintenance

AEP Process Bath General Maintenance

6.1. AEP 32 & AEP 100 process baths are controlled by regular analysis and controls. If the content of the various constituents falls below the required limits, the necessary additions should be made as outlined herein.

6.2. During periods of heavy solution usage, the analysis and controls outlined below may be required more frequently than the minimum requirement specified herein.

6.3. The temperature and humidity of the AEP coating room must be carefully controlled and the tanks must be kept permanently covered to reduce the evaporation rate of the solvent based process baths and prevent water pick up.

6.4. AEP process baths must be continuously agitated by pneumatically operated stirrers to keep the pre-alloyed metal powders in a homogenous suspension and evenly dispersed throughout the media. The tanks must be kept up to the required level by the regular addition of base solvent.

6.5. If AEP process baths cease to operate effectively, even with regular additions and maintenance, they should be discarded and replaced.

6.6. Record chemical analysis results, any additions and any tank changes electronically on MS Excel spreadsheet for for AEP process bath analysis records.

7.0 Chemical Analysis Methods

7.1. AEP Process Bath Chemical Analysis – Gravimetric & SG

This section covers gravimetric analysis of total alloy powder content, zein content and SG of process bath solvent. See paragraph 7.2. for full quantitative analysis of alloy powder content and cobalt nitrate hexahydrate by atomic absorption spectrometer (AAS).

7.1.1. Gravimetric analysis of total alloy powder content.

7.1.1.1. For each AEP process bath to be checked pre-weigh 3 off aluminium sample dishes.

7.1.1.2 Take three 10 ml samples from each AEP process bath, place a 10 ml graduated pipette (or graduated syringe) as deep as possible into the solution whilst being agitated, quickly withdraw the sample and quickly transfer the 10.0 ml into each of the three pre-weighed dishes.

7.1.1.3 Evaporate the three samples to dryness using the hot plate or similar on a low/medium heat, cool and re-weigh each dish. Calculate deposit weight.

*total solids concentration (g/l) = deposit weight (g) * **100***

7.1.1.4 The total solids concentration includes aluminium, chromium, manganese [11], cobalt nitrate hexahydrate and zein.

NOTE: *[11] Mn in AEP 32 only*

7.1.1.5. The total alloy powder content (metal) can be determined by subtracting the zein concentration from the total solids concentration. The resulting value still includes cobalt nitrate hexahydrate but this concentration is insignificant (at ~ 0.1 g/l) for the purposes of this determination.

estimated total alloy powder content (g/l) =
total solids conc (g/l) – zein conc (g/l)

7.1.1.6 Record estimated total alloy powder (metal) content concentration electronically on MS Excel spreadsheet for AEP process bath analysis records.

7.1.1.7 If required make additions of pre-alloyed powder as outlined below, para 8.0.

7.1.2. Solution SG and zein concentration.

7.1.2.1. Apparatus required:

Centrifuge
Centrifuge tubes (4 off)
Hydrometer (0.750 - 1.000)
Balance (at least 0.01g resolution)
Hot plate or similar
100 ml tall measuring cylinder
Disposable foil evaporation dish (3 off)
10 ml pipette

7.1.2.2 Take approximately 110 ml from each AEP process bath to be checked, transfer equal amounts into each of four centrifuge tubes (ensure each tube has exactly the same level of liquid for centrifuge to balance).

7.1.2.3. Centrifuge at 2400 rpm for 15 minutes; if centrifuge indicates an imbalance check liquid level in tubes and adjust as necessary. Centrifuged liquid should be a dark straw colour with all powder solids at the bottom of tube. Decant liquid to a tall 100 ml measuring cylinder.

7.1.2.4. Check SG using hydrometer. If necessary adjust solvent balance as outlined below, para 8.0.

7.1.2.5. Pipette 10 ml of the above centrifuged sample into each of three pre-weighed evaporation dishes. If required, retain remainder of sample for water content analysis, para 7.3.

7.1.2.6. Evaporate the three samples to dryness using the hot plate or similar on a low/medium heat, cool and re-weigh each dish. Calculate deposit weight.

*zein concentration (g/l) = deposit weight (g) * **100***

7.1.2.7. Record SG and zein concentration electronically on MS Excel spreadsheet for AEP process bath analysis records.

7.1.2.8. If required, make additions of zein or base solvents as outlined below, para 8.0.

7.2. AEP Process Bath Chemical Analysis - AAS

This section covers full quantitative analysis of alloy powders (Al, Cr & Mn) and cobalt nitrate hexahydrate using a PerkinElmer (PE) AAnalyst 200 atomic absorption spectrometer (AAS).
Some details may be subject to change as new AAS techniques are developed.

7.2.1. Apparatus and reagents required:

1000 mg/l (ppm) aluminium AAS standard solution
1000 mg/l (ppm) chromium AAS standard solution
1000 mg/l (ppm) manganese AAS standard solution
1000 mg/l (ppm) cobalt AAS standard solution
Concentrated hydrochloric acid (GPR RECTAPUR or eqv. grade)
Concentrated nitric acid (GPR RECTAPUR or eqv. grade)
AAS grade DI water [12]
Atomic absorption spectrometer
PE ‘Lumina’ hollow cathode lamp - multi-element (Cr, Mn & Co)
PE ‘Lumina’ hollow cathode lamp - aluminium
Hot plate
200 ml volumetric flasks [13]
100 ml volumetric flasks [13]
50 ml volumetric flasks [13]
250 ml conical flasks, 3 off
50 ml graduated measuring cylinder
10 ml pipette
5 ml pipette
10 ml graduated pipette [14]
Graduated syringe (10 ml) [14]
5 ml graduated pipette

[12] Standard de-ionised or demineralised water can be used but AAS grade preferred
[13] Quantity of flasks required may vary depending on availability and/or usage
[14] Alternatives, can use a 10 ml graduated pipette or a graduated syringe (10 ml) to withdraw sample from AEP bath

7.2.2. The results obtained by using the AAS method are directly related to the accuracy of the standards and solutions used. Great care should be taken in the preparation of these standards and solutions to ensure accuracy and consistency of results. Ensure that all the glassware used is thoroughly clean and dry before use; also ensure that all the glassware is clearly labelled identifying its contents using a black permanent marker.

7.2.3 Make up the following mixed element standards in clearly labelled 200 ml volumetric flasks using the required amount of the appropriate 1000 mg/l (ppm) standard solution for Al, Cr, Mn & Co. For chromium, cobalt and manganese make up 100 mg/l (ppm) solutions by accurately pipetting 5 ml of the 1000 mg/l (ppm) standard solutions into clearly labelled 50 ml volumetric flasks, 1/10 dilution. Alternatively pipette 10 ml into 100 ml volumetric flasks to achieve the 1/10 dilution factor. Make up to volume using an ~ 1% (vol) nitric acid solution in AAS grade DI water. See Table 3.

Where *1 ml of 1000 mg/l (ppm) standard solution in 200 ml = 5 mg/l (ppm)*

1 ml of 100 mg/l (ppm) standard solution in 200 ml = 0.5 mg/l (ppm)

TABLE 3

AAS Standard Solutions Make Up

Standard	Element	Standard solution required in 200 ml volumetric flasks		Concentration / mg/l (ppm)
		1000 mg/l (ppm) / ml	100 mg/l (ppm) / ml	
1	Al	5.0	-	25.0
	Cr	-	5.0	2.5
	Mn	-	3.0	1.5
	Co	-	3.0	1.5
2	Al	10.0	-	50.0
	Cr	-	10.0	5.0
	Mn	-	5.0	2.5
	Co	-	5.0	2.5
3	Al	20.0	-	100.0
	Cr	-	20.0	10.0
	Mn	-	10.0	5.0
	Co	-	10.0	5.0

NOTE: *Make up to volume using an ~ 1 % (vol) nitric acid solution in AAS grade DI water. Clearly label each flask. Standard solutions will degrade with time. It is recommended that all calibrated standards be dated and replaced when necessary.*

7.2.4. Take a 10 ml sample from each AEP process bath to be analysed, place a 10 ml graduated pipette or a 10 ml graduated syringe as deep as possible into the AEP solution whilst being agitated, quickly [15] withdraw the sample and quickly transfer the 10.0 ml into clearly labelled 250 ml conical flasks (one for each bath being analysed).

NOTE: *[15] This operation needs to be carried out as quickly as possible to prevent the suspended alloy powder from settling before being transferred.*

7.2.5. Evaporate to dryness; either leave overnight in a fume cupboard or alternatively heat slowly on a hot plate at low temp to prevent loss of constituents.

7.2.6. Once dry slowly and carefully add about 10 ml concentrated hydrochloric acid to dissolve the constituents, allow to stand for about 10 minutes. Bring the solution to the boil on a hot plate for 2 - 3 minutes ensuring that the metallic constituents are thoroughly dissolved in the acid but do not allow to boil dry. Add about 10 ml AAS grade DI water and continue boiling for a further 2 – 3 minutes.

7.2.7. **INITAIL SAMPLE DILUTION FACTOR 1/10 (D1)** [16] (*for cobalt nitrate hexahydrate)*

Allow to cool and transfer contents of each conical flask into clearly labelled 100 ml volumetric flasks using a suitable funnel, ensure that all the contents of each conical flask are washed into the volumetric flasks (If necessary filter the contents of the conical flask into the volumetric flask). Make up to volume using AAS grade

DI water. This gives a 1/10 dilution factor [16] and is used directly for the determination of the cobalt nitrate hexahydrate content.

7.2.7. *(continued)*

NOTE: *[16] Dilution factor based on initial 10 ml sample of AEP solvent solution; i.e. 10 ml diluted to 100 ml gives a 1/10 dilution factor.*

NOTE: *If full AAS elemental analysis of alloy powders (Al, Cr & Mn) is not required, i.e. only cobalt nitrate hexahydrate content required, there is no requirement to make further sample dilutions as outlined in paragraphs 7.2.8. & 7.2.9.*

7.2.8. **SECONDARY SAMPLE DILUTION FACTOR 1/200 (D2) [17]** *(for aluminium, Al) IF REQUIRED*

Pipette a 10 ml aliquot from each 100 ml volumetric flask into clearly labelled 200 ml volumetric flasks (alternatively pipette 5 ml into clearly labelled 100 ml volumetric flasks) to achieve a 1/20 dilution factor. Make up to volume using AAS grade DI water. This gives an overall 1/200 dilution factor [17] and is used for the determination of aluminium (Al) content.

NOTE: *[17] Overall dilution factor, based on initial 10 ml sample of AEP solvent solution diluted by 10 (1/10 as in 7.2.7) further diluted by 20 X gives a 1/200 dilution factor.*

7.2.9 **FINAL SAMPLE DILUTION FACTOR 1/2000 (D3) [18]** *(for chromium, Cr & manganese, Mn) IF REQUIRED*

Pipette a 10 ml aliquot from each 200 ml (or 100 ml) volumetric flask into clearly labelled 100 ml volumetric flasks to achieve a 1/10 dilution factor. Make up to volume using AAS grade DI water. This gives an overall 1/2000 dilution factor [18] and is used for the determination of chromium (Cr) and manganese (Mg) content.

NOTE: *[18] Overall dilution factor, based on initial 10 ml sample of AEP solvent solution diluted by 10 (1/10 as in 7.2.7), further diluted by 20 X (1/20 to give 1/200 as in 7.2.8.), finally further diluted by 10 X gives a 1/2000 dilution factor*

7.2.10. Set up and operate the PE AAnalyst 200 AAS IAW the equipment user manuals. Use the parameters below for each element to be analysed. Optimise the AAS and calibrate as shown for each of the prepared standards. For each sample take at least three readings from the AAS (default setting). For each constituent, record the mean concentration in g/l (see calculation for cobalt nitrate hexahydrate). For Al, Cr & Mn calculate the total alloy powder (metal) content (**M**) and weight %. Record all results electronically on MS Excel spreadsheet for AEP process bath analysis records.

Concentration equivalent of element in original sample (g/l) =
Dilution factor X standards concentration (mg/l) / 1000

7.2.11. AAS Parameters and standards for:

Cobalt	***Co***	*Sample dilution factor* ***1/10***
Manganese	***Mg***	*Sample dilution factor* ***1/2000***
Chromium	***Cr***	*Sample dilution factor* ***1/2000***
Aluminium	***Al***	*Sample dilution factor* ***1/200***

Cobalt Nitrate Hexahydrate $Co(NO_3)_2.6H_2O$ content:

AAS parameters	Lamp	Cobalt (or multi-element)
	Lamp current	30 mA (auto)
	Wavelength	240.7 nm
	Slit width	1.8 / 1.35 (auto)
	Flame	Air-Acetylene

Calibrate as shown   Sample dilution factor 1/10

STD 1	1.5 mg/l	rep.	**0.074** g/l $Co(NO_3)_2.6H_2O$ (0.015 g/l Co)
STD 2	2.5 mg/l	rep.	**0.123** g/l $Co(NO_3)_2.6H_2O$ (0.025 g/l Co)
STD 3	5.0 mg/l	rep	**0.246** g/l $Co(NO_3)_2.6H_2O$ (0.050 g/l Co)

NOTE: *Molecular weight of $Co(NO_3)_2.6H_2O$ is 291g of which 59g is cobalt;*
1g Co represents 59/59 + 232/59 (1 + 3.93) (i.e. Co + $(NO_3)_2.6H_2O$)
1g Co = 4.93g $Co(NO_3)_2.6H_2O$

Manganese Mn content:

AAS parameters	Lamp	Manganese (or multi-element)
	Lamp current	30 mA (auto)
	Wavelength	279.5 nm *or* 403.1 nm
	Slit width	1.8 / 0.6 (auto)
	Flame	Air-Acetylene

Calibrate as shown   Sample dilution factor 1/2000

STD 1	1.5 mg/l	represents	**3.000** g/l
STD 2	2.5 mg/l	represents	**5.000** g/l
STD 3	5.0 mg/l	represents	**10.000** g/l

Chromium Cr content:

AAS parameters	Lamp	Chromium (or multi-element)
	Lamp current	30 mA (auto)
	Wavelength	357.9 nm *or* 429.0 nm
	Slit width	2.7 / 0.8 (auto)
	Flame	Nitrous oxide-Acetylene

Calibrate as shown   Sample dilution factor 1/2000

STD 1	2.5 mg/l	represents	**5.000** g/l
STD 2	5.0 mg/l	represents	**10.000** g/l
STD 3	10.0 mg/l	represents	**20.000** g/l

Aluminium Al content:

AAS parameters	Lamp	Aluminium
	Lamp current	25 mA (auto)
	Wavelength	309.3 nm *or* 396.2 nm
	Slit width	2.7 / 0.8
	Flame	Nitrous oxide-Acetylene

Calibrate as shown Sample dilution factor 1/200

STD 1	25.0 mg/l	represents	**5.000** g/l
STD 2	50.0 mg/l	represents	**10.000** g/l
STD 3	100.0 mg/l	represents	**20.000** g/l

7.2.12. If required make additions of alloy powders and / or cobalt nitrate hexahydrate to AEP process baths, see para 8.0.

7.3. AEP Process Bath Chemical Analysis – Water Content

7.3.1. Send samples to an approved external laboratory for water content analysis.

7.3.2. Approx 50 ml sample required in a sealed plastic test tube.

7.3.3. Recommended laboratory (may use other approved laboratory if necessary):

Spectro
Palace Gate
Odiham
Hampshire RG29 1NP

Tel 01256 704000

Email enquiries@spectro-oil.com
Website www.spectro-oil.com

7.3.4 Retain laboratory reports and record results electronically on MS Excel spreadsheet for AEP process bath analysis records.

7.3.5. If the water content exceeds 2% (wt), the AEP process bath should be withdrawn from use pending further investigation and corrective action. If necessary the solution must be disposed of and replaced with a fresh solution.

8.0 Corrections & Additions

AEP Process Bath – Additions & Corrections

Refer also to AEP Process Bath Preparation, paragraph 5.0.

8.1. Calculate the additions of pre-alloyed metal powder :-

pre-alloyed metal powder required (g) =
*additional metal content required (g/l) x tank volume **T** (l)*

where :

additional metal content required (g/l) =
*specified total metal content [19] (g/l) – actual total metal content **M** (g/l)*

NOTE: *[19] concentration of total alloy powder (metal) required:*

AEP 32	*15 – 50 g/l*	*(optimum, 30 – 35 g/l)*
AEP 100	*14 – 50 g/l*	*(optimum, 20 – 25 g/l)*

For **AEP 32** process baths use Praxair **AL-123** pre-alloyed Al/Cr/Mn powder and for **AEP 100** process baths use Praxair **AL-131** pre-alloyed Al/Cr powder.
Add the required amount of powder carefully into the agitated tank by lightly sprinkling into the solution, allow about an hour before using the solution.

8.2. If the cobalt nitrate hexahydrate ($Co(NO_3)_2.6H_2O$) content is below the required limits calculate the amount to add:

cobalt nitrate hexahydrate required (g) =
*{Co nitrate req'd (g/l) – Co nitrate content (g/l)} x tank volume **T** (l)*

Dissolve the required amount of cobalt nitrate hexahydrate in the base solvent (***STEP 1***) using the guidelines outlined above (***STEP 3***). Ensure that all the cobalt nitrate is dissolved before adding to the AEP tanks.

NOTE: *Ensure that the base solvent used for this purpose is free of any zein before dissolving the cobalt nitrate hexahydrate.*

NOTE: *Ratio of cobalt nitrate hexahydrate (g/l) / zein (g/l) should be as close to 15 as possible.*

8.3. If the zein content is below the required limits calculate the amount to add, use zein solution prepared in ***STEP 2*** above.

volume of zein solution required (l) =
*wt zein required (g) / zein solution concentration **z** (g/l)*

wt zein required (g) =
*{conc of zein req'd [19] (g/l) – actual zein content (g/l)} x tank volume **T** (l)*

[19] concentration of zein required:

AEP 32	*2.00 – 3.00 g/l*
AEP 100	*1.70 – 2.30 g/l*

8.3. *(continued)*

NOTE: *It may be necessary to remove AEP process solvent from the tank, if this is the case any addition of zein solution may dilute the concentration of pre-alloyed powder and cobalt nitrate hexahydrate in the AEP bath. The extent of this dilution should be estimated and if required further additions made to replenish the solution.*

NOTE: *Ratio of cobalt nitrate hexahydrate (g/l) / zein (g/l) should be as close to 15 as possible.*

8.4. Add the prepared base solvent (***STEP 1***) as necessary to maintain the level in the AEP process baths. If the SG is less than **0.880 g cm^{-3}** add more nitromethane, if the SG is more than **0.915 g cm^{-3}** add more isopropanol.

If the SG is outside limits, calculate current volume percentage:

Volume percent of nitromethane = (actual SG – 0.785) / 0.00355

Volume percent of isopropanol = 100 – volume percent of nitromethane

8.5. Record details of any additions electronically on MS Excel spreadsheet for AEP process bath analysis records.

9.0 Disposal

Handle, store and dispose of waste AEP process bath solvents IAW Fleetlands local procedures for flammable solvents and hazardous waste.

10.0 Additional Requirements

Green coat analysis

10.1. Green coat analysis is only required for troubleshooting purposes since certified pre-alloyed metal powders are used in both AEP 32 & AEP 100 process baths. The visual appearance and average weight gain per batch are determined during the component processing operation.

10.2. If this analysis is required; from each batch of components processed, scrape off about 0.25 g of 'green coating' (prior to diffusion) from a processed part. Dissolve the 0.25 g of green coating in 10 ml concentrated hydrochloric acid in a 250 ml conical flask, stand for about 10 minutes, bring to the boil on a hot plate and proceed as outlined in paragraph 7.2. above using the AAS method to determine the relative % (wt) of the constituent metals.

10.3. The specification limits are as outlined in Table 1, AEP Process Baths & Green Coat Details.

END.