

Test Method for Catalytic Carbon Brake Disk Oxidation

RATIONALE

The current generation of runway deicers based on alkali metal acetates and formates have been shown to have a major negative effect on the oxidation resistance of carbon-carbon heat sinks in aircraft brakes. No standardized test currently exists to assess the impact of runway deicers on carbon heat sink oxidation. The test method defined in this AIR offers a quantitative procedure to evaluate the relative effect of various runway deicers on carbon brake disks.

INTRODUCTION

Runway deicing chemicals are currently approved with reference to specifications AMS1431 & AMS1435. Currently these specifications do not consider the issue of runway deicer compatibility with carbon-carbon composite friction materials extensively used in aircraft brakes. Carbon is hygroscopic and porous, and therefore readily absorbs liquids and contaminants. Since these chemicals were first introduced in the early 1990s chemical analysis of worn brakes has shown increasing contamination with alkali metal for operators with route structures which encounter icing conditions. Contamination with alkali metal catalysts increases the rate of oxidation for a given temperature exposure to a point where the degree of oxidation may be significant even for brakes subjected to typical service conditions. The impacts of oxidation can be severe from loss of refurbishment yield to loss of friction area and reduction in disc strength to a point where drive tenons or drive lugs break and the performance of the brake can be impacted. There have been growing demands from operators and airworthiness authorities for a test to assess the impact of deicing chemicals on carbon oxidation.

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1. SCOPE

The scope of the test method is to provide stakeholders including fluid manufacturers, brake manufacturers, aircraft constructors, aircraft operators and airworthiness authorities with a relative assessment of the effect of deicing chemicals on carbon oxidation. This simple test is only designed to assess the relative effects of runway deicing chemicals by measuring mass change of contaminated and bare carbon samples tested under the same conditions. It is not possible to set a general acceptance threshold oxidation limit based on this test method because carbon brake stack oxidation is a function of heat sink design and the operating environment.

2. REFERENCES

The following publications form a part of this document to the extent specified herein. The latest issue of SAE publications shall apply. The applicable issue of other publications shall be the issue in effect on the date of the purchase order. In the event of conflict between the text of this document and references cited herein, the text of this document takes precedence. Nothing in this document, however, supersedes applicable laws and regulations unless a specific exemption has been obtained.

2.1 SAE Publications

Available from SAE International, 400 Commonwealth Drive, Warrendale, PA 15096-0001, TEL: 877-606-7323 (inside USA and Canada) or 724-776-4970 (outside USA), www.sae.org.

AMS1431 Compound, Solid Runway and Taxiway Deicing/Anti-Icing

AMS1435 Fluid, Generic, Deicing/Anti-Icing, Runways and Taxiways

AIR5490 Carbon Brake Contamination

2.2 Other Applicable Documents

Available from ASTM International, 100 Barr Harbor Drive, P.O. Box C700, West Conshohocken, PA19428-2959, TEL: 610-832-9585, www.astm.org.

ASTM C1179-91 Standard Test Method for Oxidation Mass Loss of Manufactured Carbon and Graphite Materials in Air

3. OXIDATION TEST METHOD FOR ANTI-OXIDANT (AO) TREATED COUPONS

3.1 Test Coupons

- a. Material: Carbon/carbon composite brake material CARBENIX[®] 4000 (C4000[®]), Honeywell Aircraft Landing Systems, 3520 Westmoor Street, South Bend, IN 46628, United States. (Note: C4000[®] was agreed by all brake manufacturers to be a suitable material which would give results representative of all carbon-carbon brake manufacturers' materials. Alternate carbon materials/sources may be available in the future.)
- b. Configuration: Right cylinder cut from production aircraft brake discs. Dimensions, 49.911 ± 0.127 mm, (1.965 ± 0.005 inch) diameter, orientated parallel to the wear face of the as manufactured production brake discs and, 5.969 ± 0.127 mm (0.235 ± 0.005 inch) long, orientated through the thickness of the disc. Note no lubricants to be used during machining. Honeywell drawing number AX-36536. Coupon weight as supplied to be 19.800±/ 0.500 g. (Weight limits based on Honeywell C4000[®]. Note alternative carbon sources will require different weight specification limits.
- c. Identification – Coupons will be supplied with unique engraved identification numbers.
- d. At all times coupons will be handled using single use vinyl or lint-free nylon gloves.
- e. Coupons will be ultrasonic cleaned, by the test house, with one of the following fluids acetone, isopropyl alcohol, or demineralised water at room temperature for 10 minutes.
- f. Coupons will be dried after cleaning at 110 °C for 2 to 4 hours prior to preliminary weigh. Dried coupons will be stored in a desiccator.

- g. Weigh and record initial weights of all coupons to 0.001g accuracy or better. Return coupons to desiccator after weighing. (Initial weight (a))
- h. Rank coupons in weight (density) order. Allocate each ranked coupon in order to alternate groups according to how many groups required such that each seeded coupon group reflects the mean and range of the total population. Minimum group size will be 8 coupons.

3.2 Generic AO mixing instructions

- a. Consult relevant Materials Safety Data Sheet (MSDS) and wear appropriate personal protective equipment.
- b. Formulation of generic Anti-Oxidant (AO) solution is as follows:
 - 50wt % Mono Aluminium Phosphate (MALP)
Composition – Recommended supplier Sigma-Aldrich Product number 06234, brand name (FLUKA).
 - Aluminium Dihydrogen Phosphate (CAS 13530-50-2) ($\geq 48\%$ $\leq 52\text{wt } \%$)
 - Water (CAS 7732-18-5) ($\geq 48\%$ $\leq 52\text{wt } \%$)
 - 50wt % Phosphoric Acid and water
Composition
 - Phosphoric acid (CAS 7664-38-2) ($\geq 85\text{wt } \%$)
 - Water (CAS 7732-18-5) ($\leq 15\text{wt } \%$)
- c. Use a minimum of 800g of generic AO solution for a group of 8 samples to be processed. Weigh out equal quantities of MALP & Phosphoric acid according to how much solution is required.
- d. Add the acid gradually to the MALP, in increments of 25% of total volume of acid to be added, stirring for two minutes after each 25% is added.
- e. Stir the solution for 5 minutes with glass rod.
- f. Make up a separate batch of generic AO solution for each set of experiments. Store the generic AO solution in a suitable container labeled with the date and appropriate hazard warnings. Use generic AO solution within one week.

3.3 Application of Anti-oxidant Protection System: Generic AO treatment

- a. Refer to the relevant MSDS before using generic AO coating. Work in a ventilated area with appropriate personal protective equipment. Pour 800g \pm 10g of solution per 8 samples to be dipped into a large flat bottomed glass vessel. Maximum batch size 16 coupons at a time.
- b. Prepare a minimum of 8 AO coated coupons per solution plus 8 additional AO coated coupons for baseline tests.
- c. Preheat coupons to 110 °C for 1 hour before AO treatment.
- d. Remove coupons from the oven and immediately (within a maximum time of 30 seconds) fully immerse coupons in AO solution in a large glass flat bottomed vessel such that coupons lie flat on the bottom of the vessel in a single layer. Fully immerse coupons for 10 minutes + 1 minute \pm 0 minutes turn them over after 5 minutes \pm 1 minute to expose all surfaces to solution. After 10 minutes + 1 minute \pm 0 minutes remove coupons and shake off excess fluid.
- e. Allow coupons to air dry at ambient temperature for a minimum of 4 hours until the wet appearance of the AO treatment is gone prior to curing.
- f. To cure AO treatment place coupons with 1 inch space all round on heat proof contamination free tray and heat coupons at a ramp rate of 100 °C \pm 15 °C/ hour to 700 °C \pm 10 °C in nitrogen. Hold at temperature for 2 hour \pm 5 minutes. Cool coupons in oven as equipment allows.

- g. Once coupons have cooled immediately remove the coupons from the oven and transfer to a desiccator.
- h. Weigh and record coupon initial weights to 0.001g accuracy or better. Return coupons to desiccator after weighing (Initial weight (b)).
- i. Calculate the percentage pick up of AO for each coupon.

$$\frac{\text{initial wt (b)} - \text{initial wt (a)}}{\text{initial wt (a)}} * 100 = \% \text{wt. pick up}$$

- j. Reject from group any coupons that do not achieve 0.75wt % generic AO pick up. If coupons are rejected from any group replace coupons until a minimum of 8 coupons have achieved threshold generic AO pick up.
- k. Record the mean percentage pick up of AO for each group of coupons.

3.4 Contamination of coupons – Various 25% w/w runway deicer solutions

- a. Deicer solution will ideally be provided at 25% w/w concentration by deicer manufacturers but it may be necessary to make up solutions from solid deicer or by diluting a concentrated liquid deicing solution. Refer to relevant MSDS before using runway deicer solution. Wear the specified personal protective equipment.
 1. If solid deicing chemical provided dissolve “x”g of solid deicing chemical in “3x” g of demineralised water. For example 200g of solid deicer in 600g of demineralised water will provide ~0.6L of 25% w/w deicer solution.
 2. If a concentrated liquid deicing solution is supplied > 25w/w % concentration. Dilute with the appropriate weight of demineralised water to achieve a 25w/w% solution according to the following formula.

$$\text{Wt DEMIN} = (\text{Wt CONC} * \text{w/w\% CONC}) * 3 - \text{Wt CONC} * (1 - \text{w/w\% CONC})$$

Where Wt DEMIN = Weight in grams of demineralised water required to achieve dilution to 25% w/w.

Wt CONC = Weight in grams of concentrated deicer solution.

w/w% CONC = weight for weight concentration of deicer concentrate supplied expressed as a fraction.

- 3. Dilute required weight of concentrated deicer solution with appropriate weight demineralised water according to the above formula to give 25%w/w solution z
- b. Refer to relevant MSDS before using runway deicer solution. Wear specified personal protective equipment. Pour a minimum of 0.5 litres of 25% w/w deicer solution per 8 samples to be immersed into a large flat bottomed glass vessel.
- c. Prepare a minimum of 8 AO coated coupons per solution plus 8 additional AO coated coupons for baseline tests.
- d. Careful segregation will be maintained between deicer treated and control coupons to avoid cross contamination. Coupons will be placed on individual dried dedicated high density, high purity alumina discs.
- e. Fully immerse 8 coupons in runway deicer (25% w/w concentration) in a large glass flat bottomed vessel such that coupons lie flat on the bottom of the vessel in a single layer. Fully immerse coupons for 10 minutes + 1 minute/ - 0 minutes turn them over after 5 minutes +/- 1 minute to expose all surfaces to solution. After 10 minutes + 1 minute/ - 0 minutes remove coupons and shake off excess fluid.
- f. Dry deicer contaminated coupons in glass vessel for 2 hours at 150 °C ensuring no cross contamination by careful segregation with baseline coupons. Remove the coupon sample group and immediately transfer to a separate dedicated desiccator and cool for a minimum of 30 minutes to achieve room temperature.
- g. Weigh and record initial weights of all coupons to 0.001g accuracy or better immediately prior to test. (Initial weight (c))

NOTE - Do not allow coupons to stand in air prior to weighing. Some deicing chemicals (e.g. Potassium acetate) react with moisture in the air. After weighing return coupons to desiccator.

- h. Calculate the percentage pick up of deicer for each coupon immersed in deicer.

$$\frac{\text{initial wt (c).} - \text{initial wt (b)}}{\text{initial wt (a)}} * 100 = \% \text{wt. pick up}$$

- i. Repeat steps i to viii for each deicing chemical to be tested.
j. Record mean percentage pick up of deicer for each group of coupons.

3.5 Oxidation Testing

- a. Place each individual coupon centrally on its base in a clean refractory dish/ base on a large high temperature tray capable of withstanding prolonged exposure to 1022 ° F (550 °C) such that a minimum spacing of 1 inch all round is achieved. Alternate coupons on the tray such that both contaminated and uncontaminated (baseline) coupons are tested simultaneously.
- b. Select a calibrated still air muffle furnace which can achieve 1022 °F ± 10 °F or better (550 +/- 5 °C). Survey furnace temperature prior to test to establish suitability of equipment. All areas of the furnace must achieve the required temperature stability performance.
- c. Load sample tray into preheated furnace and position control thermocouple centrally between the coupons.
- d. Maintain furnace temperature at 1022 °F ± 10 °F or better (550 +/- 5 °C) in still air. Record temperature readings from the work load thermocouple either using a chart recorder or by periodic measurements.
- e. After 24 hours immediately remove the hot coupons with tray from the furnace and allow coupons to air cool for a minimum of 1 hour. Once coupons cooled sufficiently to allow handling transfer the coupons in a desiccator to achieve room temperature. Weigh each coupon and record coupon weight to 0.001g accuracy or better. (Final weight (d))
- f. Continue testing until all coupons are tested and weighed.
- g. Calculate the percentage oxidation weight loss for each coupon as follows:

$$\frac{\text{initial wt (b).} - \text{final wt (d)}}{\text{initial wt (a)}} 100 = \% \text{wt. loss}$$

NOTE - some of the weight loss included in final weight "d" results from thermal decomposition of the deicer. It would be possible to quantify this effect, but at the expense of complicating the method by the inclusion of an additional thermal stabilization and weighing step. The calculated weight loss, for the contaminated coupons, calculated in step "vii" is a minimum value for weight loss through carbon oxidation based on an assumption the deicer sees 100% thermal decomposition weight loss. This is considered acceptable provided that test conditions are designed such that thermal decomposition weight is small relative to the carbon oxidation weight loss in the presence of strong oxidation catalysts.

- h. Calculate the mean percentage oxidation weight loss and corresponding standard deviation for each group of coupons
- i. For each sample calculate the ratio of deicer pick up (section 3.4 (viii)) divided by AO pick up (section 3.3 (ix)). Calculate the mean of this ratio for each group of coupons.
- j. For each sample calculate normalized oxidation weight loss by dividing the oxidation weight loss (section 3.5 (vii)) by the ratio calculated in section 3.5 (ix).
- k. Calculate the mean normalized oxidation weight loss and standard deviation for each group of coupons.
- l. Report results for each deicer solution as follows:

Oxidation test results for AO treated coupons

Date
Test Location

Deicer name	
Deicer supplier	
Deicer concentration	
Mean pick up of deicer in coupon (%) Section 3.4 (x)	
Mean pick up of AO in coupons (%) Section 3.3 (xi)	
Mean ratio deicer pick up (%) / AO pick up (%) Section 3.5 (ix)	
Furnace temperature max (° C/ °F)	
Furnace temperature min (° C/ °F)	
Furnace temp mean (° C/ °F)	
Test time (minutes)	
Mean oxidation weight loss deicer contaminated coupons (%) Section 3.5 (viii)	
Standard deviation oxidation weight loss deicer contaminated coupons (%) Section 3.5 (viii)	
Mean normalized weight loss deicer contaminated coupons (%) Section 3.5 (xi)	
Standard deviation normalized oxidation weight loss deicer contaminated coupons (%) Section 3.5 (xi)	
Mean weight loss control coupons (%) Section 3.5 (viii)	
Standard deviation weight loss control coupons (%) Section 3.5 (viii)	

Use one table for each deicer tested.

4. NOTES

- 4.1 A change bar (I) located in the left margin is for the convenience of the user in locating areas where technical revisions, not editorial changes, have been made to the previous issue of this document. An (R) symbol to the left of the document title indicates a complete revision of the document, including technical revisions. Change bars and (R) are not used in original publications, nor in documents that contain editorial changes only.

APPENDIX A – TEST DATA TO SUBSTANTIATE METHOD

The method as outlined in section 3 was followed at two test locations, Akron and Coventry, for 5 sets of 8 C4000[®] samples supplied by Honeywell. The deicer samples were provided by a common source. The following graph shows results were consistent between the two test locations.

Combined chart generic AO coated C4000 catalytic oxidation data at two locations - 550deg C, 24 hours, still air (Dunlop & ABSC)

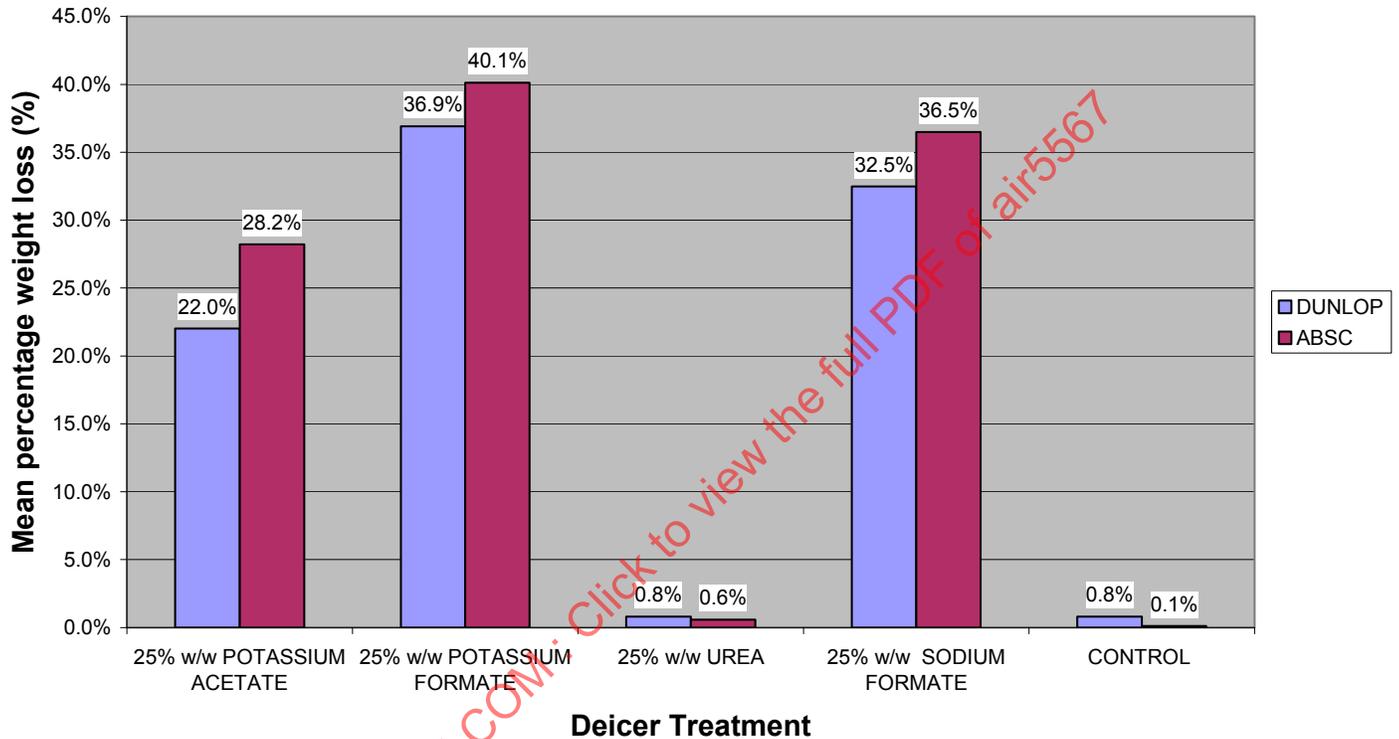


FIGURE 1 - ROUND ROBIN TEST DATA

APPENDIX B – DEVELOPMENT HISTORY OF TEST METHOD

Experimental work on development of the test method started in 2005 following a request from the G12F committee to develop a test method to assess the compatibility of runway deicing fluids with carbon-carbon composite materials used in aircraft brakes for possible incorporation into AMS1435. Preliminary discussions drew on collective experience of carbon manufacturers, published oxidation studies including a report by Hill Air Force Base HAFB and ASTM C1179-91 a published standard test method for determining oxidation mass loss of manufactured carbon and graphite materials in air. Following these discussions the following preliminary guidelines were agreed:

- Carbon test material C4000[®]
- Simple test geometry
- Bare carbon with no AO*
- Single deicer concentration
- Single temperature and time
- Testing to be carried out in still air

*Note this was later changed and final test procedure was based on carbon samples coated with a generic AO coating.

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B.1 PHASE 1 – TUNING TEST CONDITIONS FOR BARE CARBON

The Hill Air Force studies included test data for bare carbon with and without potassium acetate deicer contamination heated to 704 °C (1300 °F) for eight hours. The results showed the conditions were too severe with the uncontaminated samples exhibiting about 40% weight loss. As a result of the aggressive test conditions the test was non-discriminating with respect to deicer contamination. It is possible that oxygen demand could be limiting the reaction rate. ASTM C1179-91 advises using as low a temperature as practical to minimize scatter.

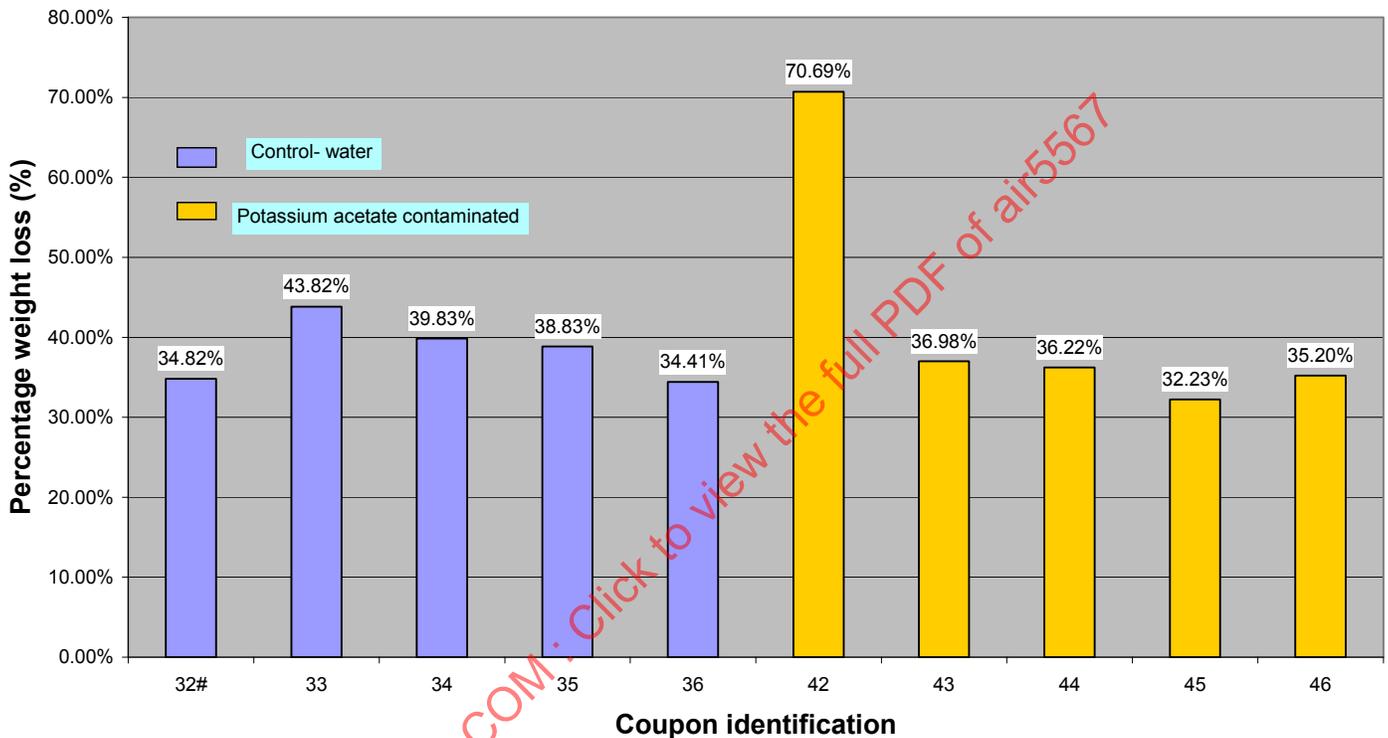
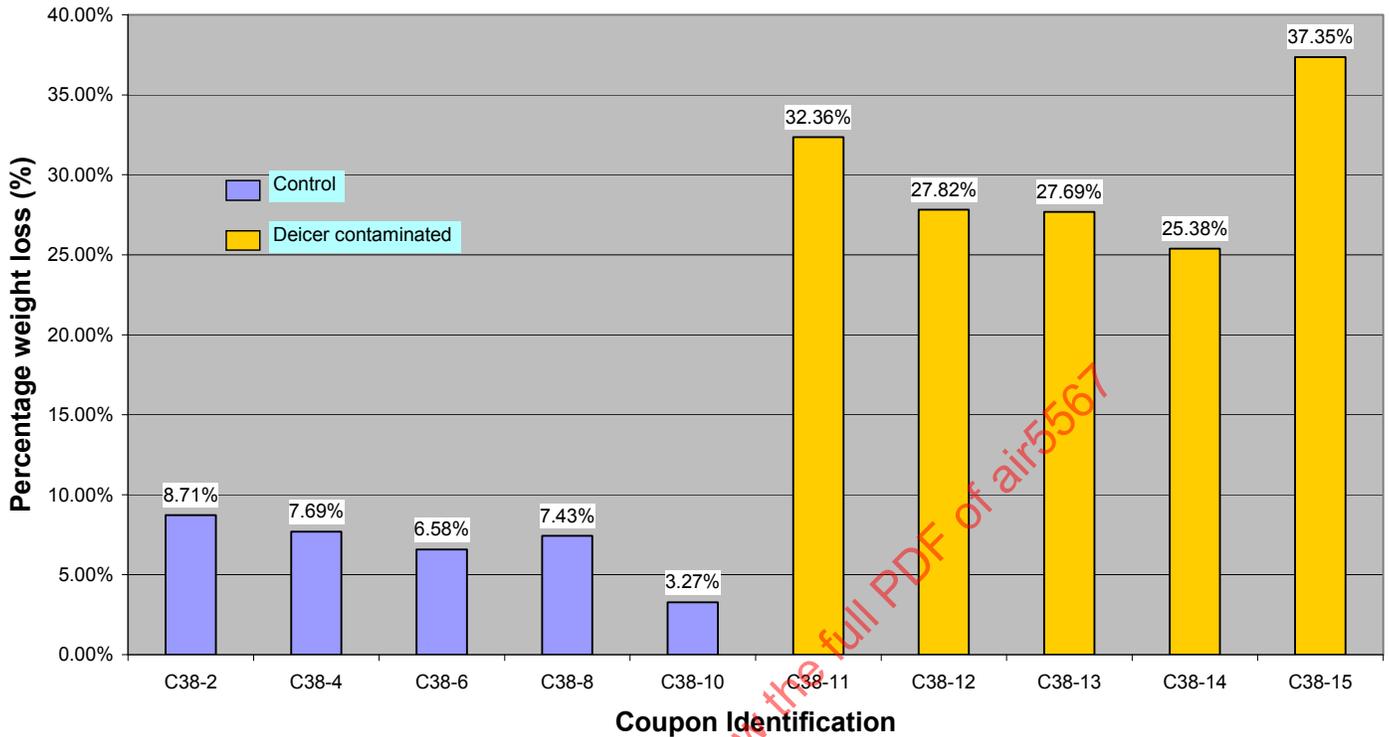
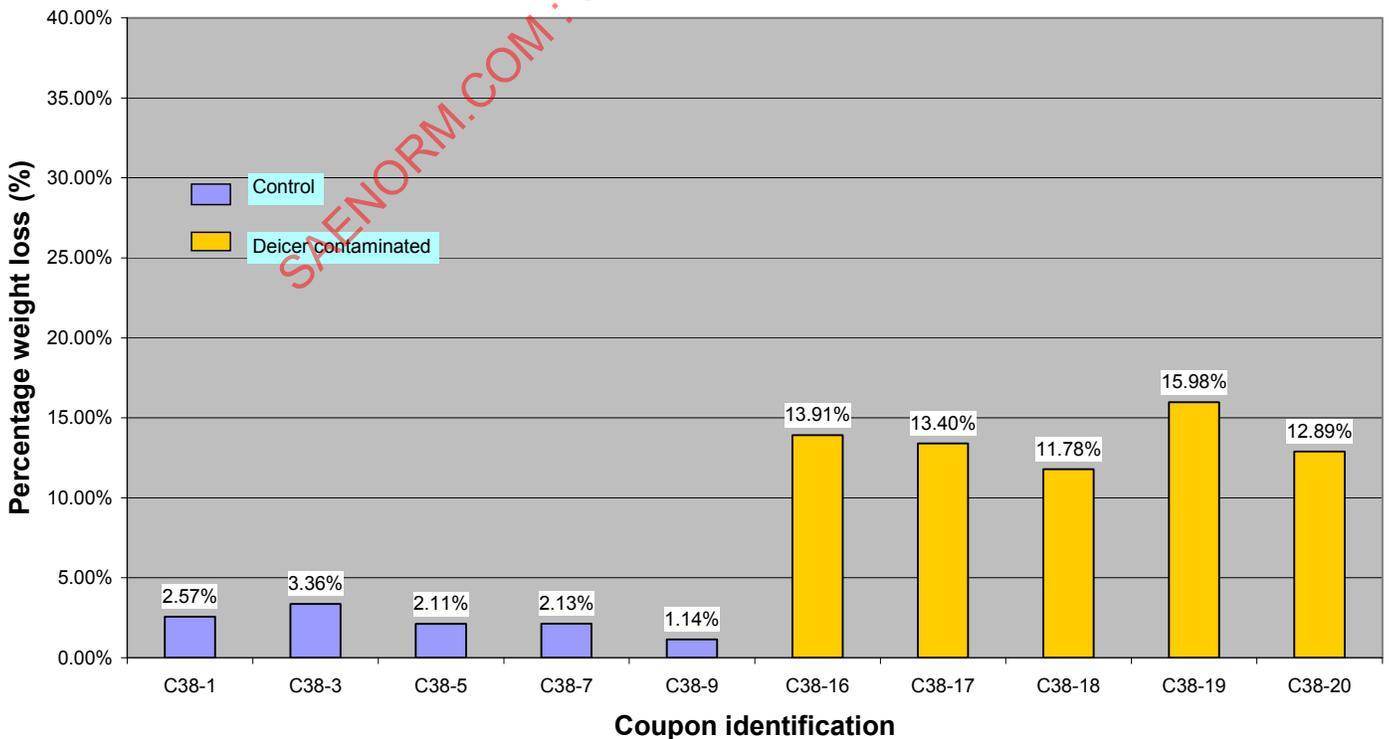
Weight loss after 700deg C , 8 hours

FIGURE 2 - HILL AIR FORCE BASE OXIDATION TEST DATA ON BARE C4000[®] CARBON AT 700 °C

Based on this information testing was proposed at reduced temperatures. Tests were carried out in October 2005 on bare C4000[®] samples both with and without potassium acetate contamination at 600 °C and 550 °C. These test results were reported in A5-A Meeting Orlando October 2005.

Weight loss after 600deg C, 4 hoursFIGURE 3 - COVENTRY OXIDATION TESTS ON BARE C4000[®] CARBON AT 600 °C**Weight loss after 550deg C, 6 hours**FIGURE 4 - COVENTRY OXIDATION TESTS ON BARE C4000[®] CARBON AT 550 °C