National Plan Oil Spill Dispersant

Effectiveness Field Test Kit (Nat-DET)

Operational Guide

Revision: June 2012

Contents

PURPOSE

The purpose of the National Plan Dispersant Effectiveness Field Test Kit (Nat-DET) is to provide instructions to operators on how to undertake an effective test of possible dispersants to use in response to the spill they are dealing with. This will then provide decision-makers with more rigorous information on which to base decisions about the likely effectiveness of the various National Plan oil spill dispersants (OSDs) on the oil slicks associated with the spill they are dealing with.

The window of opportunity for the use of OSDs on slicks at sea is often very limited. Oil changes (weathers) at sea under the action of the wind and waves, and will tend to become less dispersible as the oil thickens, its viscosity increases, and its pour point increases. Therefore it is recommended that initial dispersant effectiveness testing be carried out as soon as possible (i.e. within 6 hours following the spill).

This Operational Guide has been designed to be fast and easy to use in the field, while still providing results that are meaningful to response planners. However, it is advisable that responders practice the techniques described herein to ensure they are familiar with and competent in the use of the kit.

> **NOTE:** ***If you are unfamiliar with the Nat-DET Kits and Tests, AMSA recommends the Quick Effectiveness Test only.***

Further technical information and support

Further information about oil weathering and dispersant use can be found at www.amsa.gov.au or at: http://www.amsa.gov.au/Marine_Environment_Protection/National_plan/General_Information/Dispersants_Information/index.asp or email: SSC@amsa.gov.au.

If in doubt, please check with your State/NT technical adviser, or call AMSA's Scientific Coordinator through the Rescue Coordination Centre's **Pollution Hotline 1800 641 792** and leave your contact details, what you need and a request for the Marine Pollution Duty Officer to call you back.

INSTRUCTIONS FOR KIT CUSTODIANS

Contents and maintenance of Nat-DET Kits

ASMA expects each Nat-DET Kit custodian to check the contents of the Nat-DET Kit annually and replace equipment as needed. Page 18 below provides a copy of the inspection form. It is the responsibility of each kit custodian to replace any items damaged or lost from the kits. Replacement equipment should be identical or as similar to original as possible to ensure uniformity.

Number	Item
2	Cylindrical glass separating funnels with stopper 250ml
1 box	100 Clean-Touch gloves
1	Check Temp C thermometer (and spare replacement alkaline battery LR44 (1.5V, 175mAh, 11.6 by 5.4mm)
10	Pyrex small vials (limited numbers of extras are available on request from AMSA)
2	150ml beakers (plastic)
1	250ml beaker (plastic)
2	Erlenmeyer 250ml flask
2	25ml graduated cylinder
1	1ml glass syringe (no needle) and spare 3ml plastic syringes
1	5ml glass syringe (no needle)
1	Sample collection container

NOTE – Other materials we strongly advise to be kept with the Nat-DET Kit are:

- Fresh oil spill dispersant samples (about 100ml of each in the local stockpile and ideally less than 1 year old) and their Material Safety Data Sheets (MSDSs)
- Solvent (standard clear Mineral Spirit or 100% hydrocarbon Paint Thinner)
- Adhesive labels
- Marker pens (permanent ink)
- Cleaning fluids and tissues/cloth or other cleaning materials
- Oil and solvent resistant plastic bags for disposing of rubbish
- Small plastic funnel
- Bucket – for collecting and holding seawater samples

Personal Protective Equipment must be available with each kit, including:

- Safety glasses and disposable overalls

Most local scientific and medical equipment suppliers are able to replace any glassware or equipment that is damaged or lost. Alternatively you can contact AMSA Marine Environment Pollution Response for advice on replacing stocks, at email SSC@amsa.gov.au

Extraction solvent

Kits have not been supplied with extraction solvents for the semi-quantitative tests as these are dangerous goods and may pose a problem when trying to transport kits. The preferred solvent

(standard clear Mineral Spirit or 100% hydrocarbon Paint Thinner) can be readily purchased at most hardware stores and paint suppliers. Do NOT use petrol as an extraction solvent for health and safety, and flammability issues.

Dispersants for testing should be with the Nat- DET Kit

Not all areas have the same stockpile of National Plan dispersants. Ensure the kit always has with it a full inventory of fresh (ideally less than one year old) samples of Oil Spill Dispersants (OSDs) from the stockpiles in your area. The OSD samples should be kept in airtight glass, steel or suitable plastic containers and 100ml should be sufficient for each sample. A current copy of the material safety data sheet (MSDS) for each OSD in use must be available.

Using clean equipment

Make sure the equipment kept in the Nat-DET kit is cleaned after use. Although industrial-strength detergents may be used, the test solvent may be the most effective cleaning agent for oily solutions. Paper towelling, tissues or clean rags should be made available to assist in the cleaning of glassware. Disposable gloves have been supplied to ensure solvents and oils do not contact the skin of testers. Waste containers for oil and oil/solvent wastes generated by the testing method should be purchased and disposed of as required by local regulations.

Figure 1 – Contents of National Plan Oil Spill Dispersant Field Test Kit

A: Glass separation funnels (1 each of 2 different types)

B: Plastic beakers (2 large, 2 small)

C: Thermometer and alkaline battery LR44 (1.5V, 175mAh, 11.6 by 5.4mm)

D: 5mL glass syringe

E: 1mL glass syringe

F: Glass measuring flask (1 each of 2 different types)

G: Glass measuring cylinder (1 each of 2 different types)

H: Glass test vials (x10)

I: Sample collection container

J: disposable gloves

Not shown in this figure as not part of provided kit:

1. *Dispersant (OSD) samples and MSDSs*
2. *Cleaning fluids and rags/cloths*
3. *Rubbish disposal bags*
4. *PPE – glasses and overalls*

INSTRUCTIONS FOR CONDUCTING THE EFFECTIVENESS TESTS

Important Safety Notes – before you start

1. **Avoid direct contact with and inhalation of oil, OSD and solvents.**
2. **Always wear disposable gloves.**
3. **If skin comes in contact with chemicals, wash immediately.**
4. **Use of overalls and eye goggles is strongly advised.**
5. **Read the OSD MSDS if available.**
6. **Do the tests in a well-ventilated place.**
7. **This test can be carried out in the field, but is best done on a bench space, with a sink and running water.**
8. **Get your cleaning cloths (for spills) and rubbish containers ready before you start.**

Why perform a dispersant effectiveness test?

The Nat-DET is designed to measure the likely effectiveness, now or in the near future, of OSDs on the oils spilled. It is a critical step in the dispersant decision-making process. The aim of the test is:

a) To estimate the overall effectiveness of an OSD on the oils being tested, and
b) To determine which OSD would be most effective on these oils now or in the near future..

NOTE: *remember, oils weather and change with time. You may have to repeat the test before each day's operations (or over the day) to test whether the oil present now is still dispersible. Also, the best test of whether an oil is or remains dispersible is the actual field application of dispersant coupled with reliable, accurate and timely observations by a trained and skilled (normally aerial) observer.*

The Nat-DET approach is:

a) Prepare a **test solution** of oil and dispersant in seawater
b) Prepare a **reference solution** or set of standard solutions, and
c) Comparing the test solution visually with the reference solutions to see if the dispersant is totally, partially or not effective on that test oil.

The two different Nat-DET tests

Two tests are described in the Operational Guide:

1. **Quick-effectiveness test (QET)** – to be used when decision-makers only require an 'indication' of dispersability in a short period of time. The test involves the preparation of a test solution and a single reference solution.
2. **Semi-quantitative test (SQT)** – to be used when decision makers require an estimate of the amount (percentage) of oil dispersed by that particular dispersant. The test requires the preparation of test solution and multiple reference solutions.

If a Nat-DET user has no prior experience or training in using the kit, AMSA recommends the **Quick Effectiveness Test** only. .

NOTE: If testing more than one OSD, the tests below may be duplicated by using the second separation flask, second measuring cylinder, etc. Ensure each OSD test batch is made using clean equipment. Refer to Page 4 above for information on cleaning.

Quick effectiveness test (QET) method

The quick effectiveness test may be used as a preliminary test of Oil Spill Dispersant (OSD) effectiveness, or where a result is required in a short time period. The quick test involves applying the OSD to the test oil, mixing with seawater and observing its effectiveness in dispersing oil, compared to a reference solution containing only oil and seawater. This test may be used if a simple 'pass/fail' criterion is all that is required when assessing if an OSD will be effective on particular oil.

NOTE - *If you do not have a sample of the actual spilled oil due to access or the need for a quick decision, an equivalent grade of oil may be used. However, it is important to remember that even if identical to the spilled oil, this oil will not have weathered in the same way as the spilled oil, and so your results will reflect this ("fresh" oil is always more easily dispersed). Whatever result you get, please ensure the decision-maker knows what oil you tested – identify the stand-in if you used one.*

Allow a minimum of 20 minutes for the complete test.

QET – Safety and PPE:

- Latex gloves and eye goggles
- Use of overalls is advised
- A bench and sink with running water is advised
- Cloths and rubbish containers available

QET – Equipment:

- Bucket-sized sample of fresh seawater from or near incident site – the larger the better as it will maintain sea temperature better while you do the tests – keep out of sunlight!
- A representative sample of the oil from the incident (or equivalent grade of oil – see note above)
- Oil spill dispersant(s) to be tested
- Plastic beaker
- 5ml syringe + 1ml syringe
- Separating funnel
- Vials
- Plastic funnel – use for pouring liquids

QET – preparing the test solution

1. Collect a sample of fresh, clean seawater in a large plastic beaker or bucket, measure its temperature with the digital thermometer as soon as possible and record details. (Allow the probe to settle down prior to reading temperature and keep out of direct sunlight, if possible.)
2. Label two test vials – one with ***Test and the name of the OSD***, and one with ***Reference***.
3. In a large plastic beaker, mix OSD and test oil at the correct dispersant to oil application ratio (check the manufacturer's instructions or the AMSA Dispersant Guidelines as referred to on page 3 above). For example if 1:20 ratio is recommended add 1 ml of OSD (1 ml syringe) to 20 ml of oil (measuring cylinder). Add the OSD drop-wise as evenly as possible over the surface of the oil. Stir the solution briefly to ensure homogeneity.
4. Close the valve at the bottom of the separating funnel (it should be horizontal) then measure 100 ml of seawater into the funnel. Using the 5 ml syringe, extract 2 ml of the oil/dispersant mixture and add to the seawater in the funnel. Ensure the stopper is tight in the top of the separating funnel.
5. Mix the oil/OSD/seawater solution by slowly rotating the separation funnel end over end (at a rate of about one half turn for every 2 seconds) for exactly 2 minutes (keep a thumb or finger over the stopper to ensure it remains tight and does not leak). This mimics the mixing action of the ocean on the OSD/oil mixture.
6. Stand the separating funnel upright for 5 minutes, removing the stopper from the top of separating funnel. This allows undispersed oil to separate from the water/dispersed oil layer.
7. Using the plastic valve at the bottom of the separating funnel, carefully drain 10 ml of the dispersed oil/water mixture (lower layer in the separating funnel) into a test vial already labelled as ***Test and the name of the OSD***.

NOTE: *If testing alternative oil spill dispersants, repeat steps 2 – 7 for each dispersant type making sure you have already labelled enough test vials. Clean the equipment thoroughly between each test (refer to page 3 above for cleaning). These solutions represent the amount of oil successfully dispersed by each OSD, and can be visually compared to determine the effectiveness of each OSD.*

QET – Preparing the reference solution

The reference solution of non-dispersed oil and seawater is to compare with your test mixture.

1. Mix approximately 2ml of test oil with 100ml of seawater in the (clean) separation flask.
2. Mix the oil and seawater solution by slowly rotating the separation funnel end over end (at a rate of about one half turn for every 2 seconds) for exactly 2 minutes (keep a thumb or finger

over the stopper to ensure it remains tight and does not leak). This mimics the mixing action of the ocean on the oil mixture.

3. Stand for 5 minutes before draining 10mL of the solution into the test vial labelled as ***Reference***. This solution should be relatively clear due to the insolubility of oil in seawater.

QET – Interpretation

Visually compare the ***Test Mixture***(s) with the ***Reference*** solution containing just oil and seawater. If the OSD is effective, you will see the test mixtures will be darker or more opaque than the water in the reference mixture (see Figure 2 below) due to the quantity of dispersed oil suspended in the seawater.

Figure 2 – QET results - comparing the Reference with the Test/OSD

The semi-quantitative test (SQT) method

The semi-quantitative test is more time-consuming, complex and exacting, involving two different processes to produce a test solution and a series of reference solutions to compare with the test solution. These will give partial and full dispersion reference sets (i.e. 25%, 50%, 75% and 100%) to compare with the test solution. It also requires the use of solvents to assist the oil/SOD mixtures to remain in suspension to assist visual comparison and is temperature dependent.

NOTE - *If you do not have a sample of the actual spilled oil due to access or the need for a quick decision, an equivalent grade of oil may be used. However, it is important to remember that even if identical to the spilled oil, this oil will not have weathered in the same way as the spilled oil, and so your results will reflect this ("fresh" oil is always more easily dispersed). Whatever result you get, please ensure the decision-maker knows what oil you tested – identify the stand-in if you used one.*

Allow a minimum of 60 minutes for the complete test.

SQT – Safety and PPE:

- Latex gloves and eye goggles
- Use of overalls is advised
- A bench and sink with running water is strongly advised
- Cloths and rubbish containers available

SQT Part 1 – Preparing the test mixture(s)

Equipment:

- Oil spill dispersant(s) to be tested
- A representative sample of the oil from the incident (or equivalent grade of oil – see note above)
- Bucket-sized sample of fresh seawater from or near incident site – the larger the better as it will maintain sea temperature better while you do the tests – keep out of sunlight!
- 5ml syringe
- 1ml syringe
- Measuring cylinder
- Measuring flask (bottle)
- Plastic beaker
- Separating funnel
- Thermometer
- Solvent (not supplied with dispersant test kit)
- Plastic funnel – use for pouring liquids

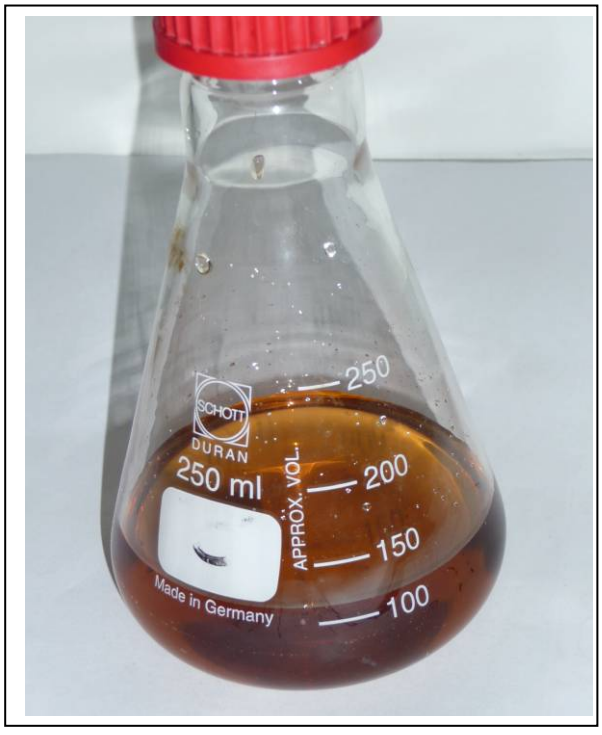
Preparing the test mixture(s)

1. Collect a sample of fresh, clean seawater in a large plastic beaker or bucket, measure its temperature with the digital thermometer as soon as possible and record details. (Allow the probe to settle down prior to reading temperature and keep out of direct sunlight, if possible.)
2. Label another large plastic beaker **Mixture A.** In this plastic beaker, apply dispersant to a sample of the test oil at the correct OSD to oil application ratio. For example if this is 1:20 add 1 ml of dispersant (1 ml syringe) to 20 ml of oil (measuring cylinder). Add the OSD drop-wise as evenly as possible over the surface of the oil. Stir solution briefly. (Do NOT add any seawater). Carefully put this beaker of **Mixture A** to one side – you will also need it for Part 2. (See Figure 3).
3. Check the temperature of the seawater sample is still similar to when it was first collected (i.e. sea state temperature). Close the valve of the separating funnel (it should be horizontal) then measure 100 ml of seawater into the funnel.
4. Using the 5ml syringe, add 2 ml of **Mixture A** to the surface of the seawater and fasten stopper onto the separating funnel. Ensure the separating funnel valve is closed (valve tap is horizontal)
5. Mix the oil/OSD/seawater solution by slowly rotating the separation funnel end over end (at a rate of about one half turn for every 2 seconds) for exactly 2 minutes (keep a thumb or finger over the stopper to ensure it remains tight and does not leak). This mimics the mixing action of the ocean on the OSD/oil mixture. (See Figure 3).
6. Stand the separating funnel stand upright for 5 minutes, removing the stopper from the top of separating funnel. This allows undispersed oil to separate from the water/dispersed oil layer.
7. Label a measuring cylinder **Mixture B** and a measuring flask (bottle) **Mixture C.**
8. Using the plastic valve on the separating funnel, carefully drain 10 ml of the dispersed oil/water mixture (lower layer in separating funnel) into this measuring cylinder labelled **Mixture B.**
9. Dilute **Mixture B** by transferring it to a measuring flask and add 100 ml of clean solvent. Shake vigorously (ensure cap is securely screwed on) for 30 seconds, loosen the cap to release any pressure, and let to stand for 20 minutes. **Mixture C** represents the dispersed oil, in a solution of solvent to help it remain suspended for visual reference. (See Figure 3).

Figure 3 - Semi-quantitative test – Part 1 test mixture

Step 2 : Oil and dispersant mix in the beaker

Step 5: Dispersed oil in the separating funnel



Step 10: Mixture C – dispersed oil suspended in solvent with consistent colour.

SQT Part 2 – Preparing the reference or standard mixtures

Equipment

- Fresh solvent
- Mixture A
- Mixture C
- 1ml measuring syringe
- Measuring flask (bottle)
- 5 glass vials (labelled Std1:100%, Std2: 75%, Std3: 50%, Std4: 25% and Test)

Preparing the reference or standard mixtures

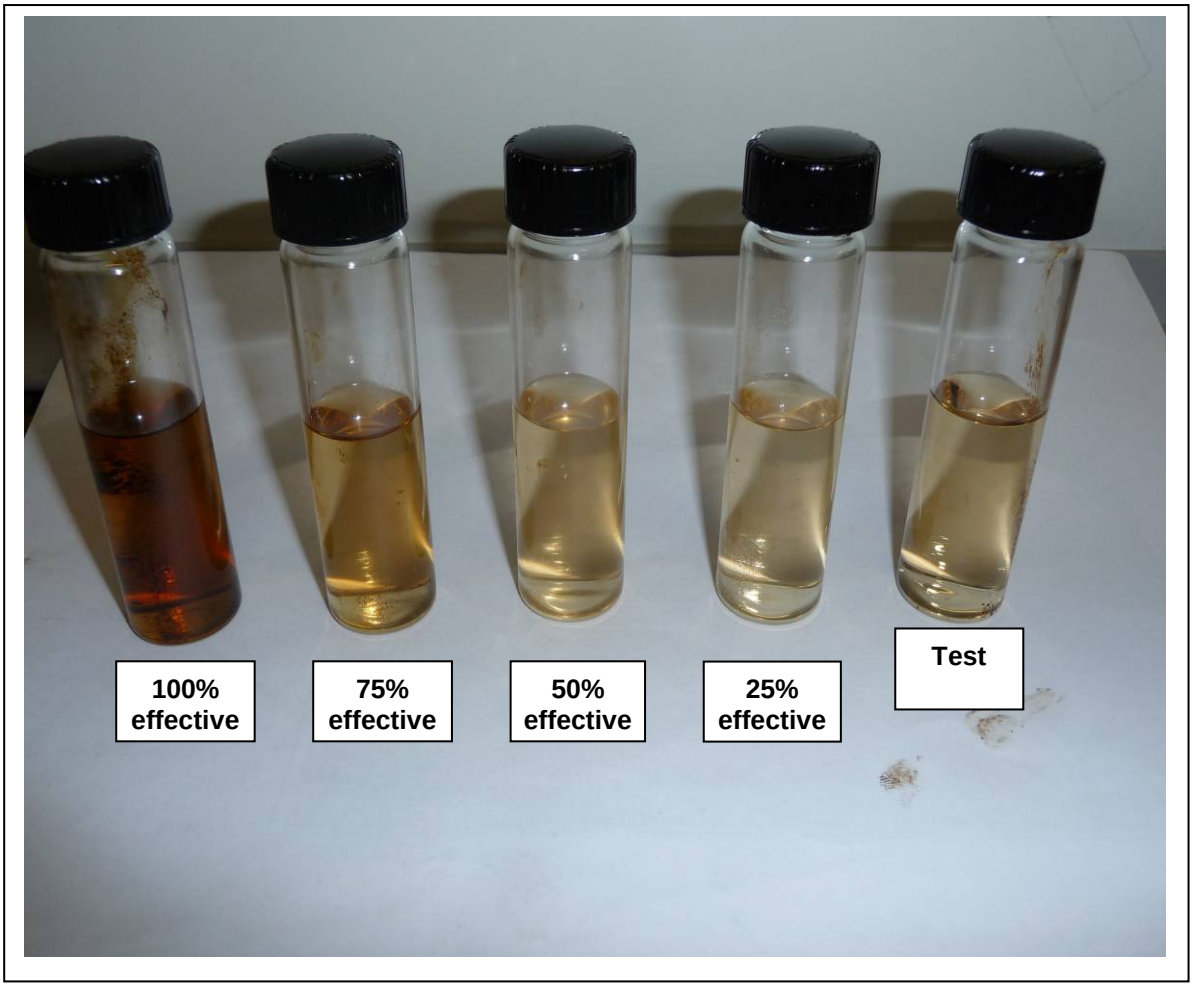
10. Prepare **Standard 1** by using the 1ml syringe to add 0.5 ml of **Mixture A** (oil/dispersant) to 250 ml of fresh solvent in the measuring flask (bottle). This represents 100% dispersion. The ratio has been calculated on the same basis as the dispersion test i.e. 2 ml oil/dispersant in 100 ml water diluted 10 times.
11. Add a small amount of **Standard 1** to the small vial labelled **Std1: 100%**.
12. To make reference solutions of lower effectiveness, dilute **Standard 1** as follows:
    - 75% effectiveness solution: mix 3 parts of **Standard 1** with 1 part fresh solvent in the measuring flask (i.e. 60ml Standard 1 with 20ml solvent). Decant this solution into the small vial labelled **Std2: 75%**.
    - 50 % effectiveness solution: Mix 1 part of **Standard 1** with 1 part fresh solvent using the measuring flask (i.e. 40ml Standard 1 with 40ml solvent). Decant this solution into the small vial labelled **Std3: 50%.**
    - 25 % effectiveness solution: Mix 1 part of **Standard 1** with 3 parts of fresh solvent (i.e. 20ml Standard 1 with 60ml solvent). Decant this solution into the small vial labelled **Std4: 25%.**
13. You should now have solutions in 4 glass vials representing dispersion effectiveness of 100%, 75%, 50% and 25% and labelled as such.
14. Add the actual dispersed oil sample (Mixture C) to the remaining small vial **(Test)** to allow you to compare this with the standards prepared.

SQT – Interpretation

Visually compare the **Test** against the **standard solutions** you have made up (see Fig 4 below) and categorise the dispersant effectiveness of the **Test.** Use approximate values if necessary, i.e. 80% effective, <25% effective. Record your results.

NOTE - *Strongly coloured oil/solvent solutions (dark oils) may require even further dilution to be compared visually. Alternatively the use of a strong light or white background behind the sample tubes can also be used. If further dilution is carried out it must be done using the same dilution ratio for all five vials (**Test** and **Stds1-4**).*

Figure 4: Semi-quantitative test (SQT)results



Step 15 and Interpretation: comparison of test solution (right) and reference solutions (in descending order of effectiveness). The test solution is equivalent to the 50% effectiveness reference solution.

REFERENCES

This technique has been based primarily on the method developed for the American Petroleum Institute (API) and correlated with the Warren Springs Laboratory (WSL) dispersant testing procedure. It is essentially a modified US EPA Field Dispersant Effective Test (FDET) - NTIS publication # PB87-234 886/AS. Further details on the accuracy and reproducibility of the method can be obtained from Publication No. API #4478 "*Rapid Test for Dispersant Effectiveness at Oil Spill Sites*" January 1989 API 1220L Street Northwest, Washington, DC 10005.

IMPROVEMENTS TO Nat-DET and Kits

The Nat-DET kits have been designed to be robust for field use, yet as simple as possible for the untrained user. The kits have also been designed to provide a reasonable degree of confidence to response planners of the dispersability of oil slicks without the use of sophisticated analytical laboratory equipment. However, if users of the Nat-DET field kits have any suggestions to improve the technique or contents of the kits please contact AMSA at SSC@amsa.gov.au.

ACKNOWLEDGEMENTS

Many thanks to Bruce Brady, Deputy ESC for Victoria until 2011, for both insightful commentary and practical tips during the review of this Operational Guide.

AMSA Document reference TRIM: D12/89666

Officer's name:…….....and State:……..

email:.....……..Date of Kit inspection:……./……/……

Reporting form for the annual Nat-DET kits contents inspection and maintenance

The kit is contained within one "Pelican" brand orange case. It is the responsibility of Nat-DET kit custodians to replace any items damaged or lost from the kits. The checklist below identifies the equipment provided with the kit.

Number	Item	Checked & status
2	Cylindrical glass separating funnels with stopper 250ml	
1 box	100 Clean-Touch gloves	
1	Check Temp C thermometer (and spare replacement alkaline battery LR44 – 1.5V, 175mAh, 11.6 by 5.4mm)	
10	Pyrex small vials (limited numbers of extras are available on request from AMSA[1])	
2	150ml beakers (plastic)	
1	250ml beaker (plastic)	
2	Erlenmeyer 250ml flask	
2	25ml graduated cylinder	
1	1ml glass syringe (no needle) and spare 3ml plastic syringes	
1	5ml glass syringe (no needle)	
1	Sample collection container	
Signature of Inspecting Officer that the information on the list above is correct:		

Other materials AMSA strongly advises to be kept with the Nat-DET Kit are:

- Fresh oil spill dispersant samples (about 100ml of each in the local stockpile and ideally less than 1 year old) and their Material Safety Data Sheets (MSDSs)
- Solvent[2] (standard clear Mineral Spirit or 100% hydrocarbon Paint Thinner)
- Adhesive labels
- Marker pens (permanent ink)
- Cleaning fluids and tissues/cloth or other cleaning materials
- Oil and solvent resistant plastic bags for disposing of rubbish
- Small plastic funnel
- Bucket – for collecting and holding seawater samples
- Personal Protective Equipment must be available with each kit, including safety glasses and Disposable overalls

Notes

1. AMSA is holding some spare equipment at the Canberra office, and where stocks exist these may be available upon request. Most local scientific and medical equipment suppliers will able to replace any glassware or equipment that is damaged or lost, otherwise please contact AMSA at SSC@amsa.gov.au.

2. To minimise issues of the transport of dangerous goods to and between the States/NT, kits have not been initially supplied with extraction solvents. Solvent can be purchased at most hardware stores and paint suppliers throughout the country.