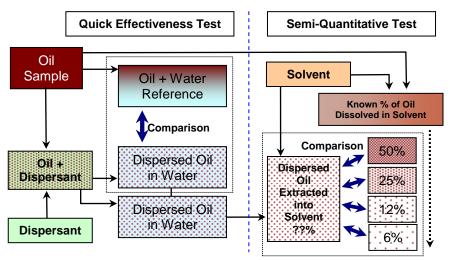


Thanks to the Queensland Department of Environment and Heritage Protection; they developed this excellent checklist to use with the <u>2012 National Plan Operational Guide</u>. Printed checklists should be kept to record findings and as confirm correct procedures were followed.

### 1 BACKGROUND

The Oil Spill Dispersant Effectiveness Test is essential for informed decision making on the potential use of dispersants. This checklist provides a summary of items needed and the main procedures to be followed. There are two tests:

- The "quick effectiveness test", conducted as an initial check to see if any of the available dispersants show signs of being effective. This test simply compares an oil and water mixture with an oil and dispersant and water mixture.
- *The "semi-quantitative test"*, carried out for those dispersants that show a significant likelihood of being effective. This test compares a solvent extraction of the dispersed oil with known percentage dilutions of the oil in solvent.



### 2 PREPARATION & EQUIPMENT

It is important that all work is carried out in well ventilated places, due to the levels of solvent and oil fumes. You will need:

- O Large bench in ventilated area with sink.
- O Waterproof overalls or apron and eye protection.
- O 1 box nitrile gloves (<u>not latex</u> gloves which are permeable to toxic hydrocarbons).
- O 1 box tissues or paper towels.
- O 1 garbage bag for solid waste and plastic sheet or newspaper to cover workbench.
- O Stand for separation funnels (sturdy cardboard box with cut holes or a retort stand).
- O 2L solvent for extractions and cleaning (white spirits or turps).
- O 10L plastic bucket for liquid wastes.

- O 15ml vials about 30 (up to 23 may be used).
- O Mark all vials with a 10ml line copied from an accurately filled 10ml reference vial.
- O Fine point permanent marker to write on vials and labels.
- O Small stick-on pre-prepared labels to mark vials.
- O Test tube rack (collapsible) or very small cardboard box with holes cut into it.
- O Blutac to stand vials up for photographs.
- O Plastic teaspoons for stirrers and 1ml & 15ml disposable syringes.
- O Safety Data Sheets for hazardous materials being handled (read carefully).
- O Dishwashing detergent and bottle brushes.

### **TIPS**

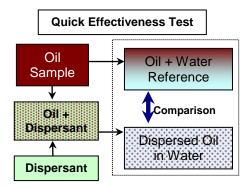
- Minimise contact of oil with glassware, to cut down on washing up.
- Add water and solvents directly to graduated glassware to save on using measuring cylinders.
- Label or mark all glassware to avoid confusion.
- Have pre-prepared labels for final photographs with dilutions and dispersant types.
- Use a sheet of white paper taped against a window as a backlit background for sample photos.
- Leave caps on containers to minimise fumes.
- Accurately fill one vial with 10ml of water and use as a standard to save on measuring cylinders.
- Mark the shaft of the syringe plunger against the top of the barrel at the right volumes prior to use, as it is difficult to read markings and see the end of the black plunger against the black oil.

### 2.1 Oil Samples

insp	esh oil sample should be obtained as early as possible after each spill, ideally by the feet the casualty or oiled waters, e.g. AMSA surveyors or maritime operations officers the ship's bunkers as well as samples from the spilled slick. You will need:	
	300ml screw top plastic containers labelled with:	
	Source of oil: (e.g. ship's bunker or oil slick on water)  Date of sampling:	

### 2

	Source of oii(e.g. ship's bunker of oii stick on water)		
	Date of sampling:		
	Time of sampling:		
2.2	Sea Water Sample		
A 1	arge sample is best to minimise temperature variation during the testing. You will need:		
	1 10L clean plastic container with lid or clean drinking water esky.		
	Digital thermometer with spare battery (or alcohol thermometer).		
	Measure the temperature of the seawater at the time of collection and label with:		
	Source of seawater:		
	Date of sampling:		
	Time of sampling:		
	Temperature: <u>°C</u>		



### 3 QUICK EFFECTIVENESS TEST

Used for a preliminary indication of dispersant effectiveness or for selection of dispersants for further testing.

The Quick Effectiveness Test is a basic simulation of what would happen in the field, that is:

- oil + dispersant + water + agitation compared to,
- oil + water only + agitation

No solvent extraction is done and only a general comparison between the samples with and without dispersant is made.

### 3.1 EQUIPMENT

For the quick effectiveness test you will need:

- 0 250ml beaker for measuring seawater.
- 0 125ml separating funnels with caps for simulating ocean mixing.
- 100ml beakers for mixing dispersant and oil (typically 3 are needed). 0
- 0 1ml plastic disposable syringes for adding dispersant.
- 2ml plastic disposable syringes for dispersant/oil mixture. 0
- 0 5ml and/or 10ml syringes for oil (up to 50ml may be needed).
- 0 Stick on labels set marked - Blank, Disp 1, Disp 2, Disp 3.
- 0 15ml vials - for samples for this test (typically 4 are needed).
- 15ml vials for dispersed oil samples held over for use in the following semi-quantitative test (Section 4 – typically 3 are needed).

Up to four sets of equipment may be needed for all the tests, i.e. blank plus three dispersants. Be sure to have plenty of the disposable items such as syringes and stirrers. If there are insufficient items of glassware, be prepared to do lots of washing up. Have solvent resistant waste bottles handy.

### 3.2 QUICK EFFECTIVENESS TEST PROCEDURE (~20 minutes)

This test prepares an <u>oil+water</u> sample and an <u>oil+dispersant+water</u> test mix. Test mixtures should be at about the same temperature as the water in the environment where the dispersant is to be applied.

.2.1	Oil + dispersant + water test mix
	Check Appendix 2 for the appropriate ratio for each available dispersant to add to the oil.
	Add 1ml dispersant to appropriate volume of oil in a 100ml beaker (typically 1:20ml or 1:25ml).
	Mix thoroughly with a plastic stirrer until the dispersant is dissolved.
	Check that the seawater temperature is similar to the temperature in the field (warm or cool as req.)
	Add 100ml of seawater to the separating funnel, close the valve first.
	Add 2ml dispersant+oil mixture to the seawater in the separating funnel, replace cap.
	Simulate ocean mixing by holding the separating funnel and rotating it end-over-end continuously
	a half turn per 2 seconds. Do that for exactly 2 minutes. Note: Tighten cap first and hold firmly in
	place.
	Stand the congrating funnal unright in a holder remove the cap allow it to cattle for 5 minutes

- Stand the separating funnel upright in a holder, remove the cap, allow it to settle for 5 minutes.
- Drain 10ml of dispersed oil solution into a calibrated vial.
- \#-\[Drain 10\text{ml of dispersed oil solution into a second calibrated vial, label and put aside for making up the sample in the semi-quantitative test (See 4.2)].
- Repeat the procedure above for all available dispersants that might be used.

3.2.2	Oil + water undispersed reference sample
	Add 2ml of untreated oil to 100ml of seawater in the separating funnel (without dispersant).
	Rotate the separating funnel end-over-end continuously <i>a half turn per 2 seconds</i> for <i>exactly 2 minutes</i> to simulate ocean mixing of the oil and water. Note: Tighten cap first and hold firmly in
	place.
	Stand the separating funnel upright in the holder, remove cap, allow it to settle for <i>5 minutes</i> .
	Drain 10ml of the oil + water mix into a 10ml calibrated vial as the undispersed reference.
	Comparison of dispersed and undispersed oil ompare and photograph the final set of blank and dispersant test vials. Record results below.
Note -	# Additional 10ml samples of dispersed oil in seawater in 15ml vials must be kept to make up the sample solutions for the semi-quantitative test for each dispersant being tested.

### 3.3 QUICK EFFECTIVENESS TEST RESULTS REPORT

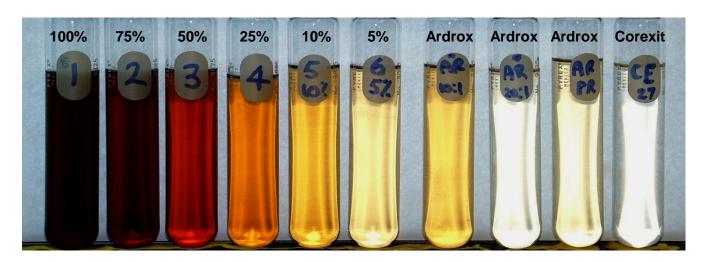
Visually compare the oil+water mixture with the oil+dispersant+water mixture.

For each dispersant, does the water phase appear to contain a significant amount of dispersed oil?

	Seawater Temperature	Ratio of oil to dispersant	Effective dispersal of oil
Oil in water only (blank) reference	°C	N/A	
Dispersant 1:	°C	to 1	☐ YES / ☐ NO
Dispersant 2:	°C	to 1	☐ YES / ☐ NO
Dispersant 3:	°C	to 1	☐ YES / ☐ NO

### **4 SEMI-QUANTITATIVE TEST**

This test should be carried out for all dispersants that showed significant effectiveness in the initial Quick Effectiveness Test. This test uses solvent to extract the oil dispersed in the seawater of the quick effectiveness test samples. The density/colour of this is then visually compared to reference samples of known percentages of the oil dissolved in solvent.



### **4.1 EQUIPMENT**

For the semi-quantitative test you will need:

- O 1ml plastic syringe for measuring out 0.5ml oil.
- O 15ml vials 100%, 50%, 25%, 12%, 6%, dispersed oil test.
- O 25ml measuring cylinder for measuring out clean solvent.
- O 25ml measuring cylinder for measuring out standard solutions.
- O 250ml conical flask with cap for preparing solvent extraction sample.
- O 250ml conical flask with cap for mixing standards.
- O Stick on labels set 2 100%, 50%, 25%, 12%, 6%, Disp1, Disp 2, Disp 3.

Note – Up to three sets of equipment may be needed for all the tests, i.e. up to three dispersants.

Be sure to have plenty of vials. If there are not sufficient items of glassware, be prepared for lots of washing up.

### 4.2 PROCEDURE - SEMI-QUANTITATIVE SAMPLE SOLVENT EXTRACTION (~30 minutes)

This procedure uses solvent to separate the dispersed oil from the oil-water mixture to compare against standards where the % of oil dissolved in solvent is known.

tanua	itus where the 70 of on dissolved in solvent is known.
	Find the vial with 10ml of dispersed oil prepared in the quick effectiveness test (See 3.2 \mathbb{H}).
	Transfer the contents of the vial to a 250ml conical flask.
	Add 100ml solvent to the conical flask. Note: Cap needs to be firmly tightened.
	Shake vigorously for 30 seconds, then loosen the cap and let it stand for 20 minutes to allow the oil
	now dissolved in the solvent to separate from the water.
	Carefully pour 10 ml of the oil+solvent phase from the top into a calibrated vial.
	Keep the vial to compare with and photograph against the relevant standard set (4.3 below).

### **4.3 PROCEDURE – STANDARDS PREPARATION** (~30 minutes)

The disperse oil extract is compared	against a set of known % oil standards to visually estimate how much
oil was dispersed in the test sample.	Except for the 100% standard the following steps are serial 1:1
dilutions.	

<b>100% standard</b> - Add 250ml solvent to 250 ml conical flask, then add 0.5ml of oil. Make sure cap is firmly tightened. (Note - This is a 1:500 dilution, which is the same as the combined dilutions of 2ml:100ml from the quick test, then 10ml:100ml from this semi-quantitative test).
<b>50% standard</b> – Add 25ml of the 100% standard to a beaker, then add 25ml solvent. Pour 10ml of this into a calibrated vial marked 50%.
<b>25% standard</b> – Add 25ml of the 50% standard to a beaker, then add 25ml solvent. Pour 10ml of this into a calibrated vial marked 25%.
<b>12% standard</b> – Add 25ml of the 25% standard to a beaker, then add 25ml solvent. Pour 10ml of this into a calibrated vial marked 12%.
<b>6% standard</b> – Add 25ml of the 12% standard to a beaker, then add 25ml solvent. Pour 10ml of this into a calibrated vial marked 6%.
<b>Visually compare</b> the relevant dispersed oil extracts with the known % concentration standards (100%, 50%, 25%, 12%, 6%) against a neutral white background (See Section 5).

### <u>4.4 SEMI-QUANTITATIVE EFFECTIVENESS TEST RESULTS REPORT</u>

From your comparison of the test sample(s) with the solvent standards, what was the estimated level of effectiveness? Ensure that the ratio of dispersant to oil, as well as dispersant effectiveness, are clearly stated.

	Oil to dispersant ratio (e.g. 20:1, 25:1, 10:1)	Estimated Dispersant Effectiveness (%)
Dispersant 1:	to 1	
Dispersant 2:	to 1	
Dispersant 3:	to 1	

### **5 PHOTOGRAPHS & REPORTING**

Samples from both the quick and semi-quantitative tests should be photographed for inclusion in reports and records.

114 1	ceords.
	Stand labelled test and standards vials up in order against a flat white background.
	White paper taped to a window will provide a neutral background and backlighting.
	Stand vials on Blutac to keep them upright and in place.
	Set the digital camera to MACRO (close-up) mode for close-up photos ( flower symbol).
	Take photos of the vials with the camera flash set to <i>Redeye</i> or <i>Fill</i> as well as $Off \otimes \mathbb{Z} \otimes .$
	Photograph the extract next to the known % standards square to the camera, not at an oblique angle

# 6 CLEANUP It is important to recondition all equipment and clean-up workspaces as soon as possible, to ensure readiness should additional testing be required. Glassware washed and dried, bench and washing up area cleaned and tidied. Wastes appropriately disposed of (solvent and oil will need to be disposed of as regulated waste). Broken items and consumables replaced (or appropriate arrangements made). All items packed away into the kit ready for immediate use. 7 SUMMARY Retain this checklist as a record of the findings of the test and to confirm that appropriate procedures were followed.

incident.		
Test completed by:	Signature:	
Date:	Time:	
NOTES:		

# APPENDIX 1: EQUIPMENT LIST

Items	Kit	Extras Box
Solvent (white spirits or turps)		nil (need to buy 2L)
10L plastic buckets		nil (need to buy 2)
(for seawater and liquid wastes)		
10L plastic buckets		nil (may need to buy 2)
(for washing up)		
Stand for separation funnels		2
Clamps and bossheads		4
Tissues, nitrile gloves		1 box each
Garbage bags		6
Dishwashing detergent		100ml
Bottle brush		1 small
Tea towels, washing cloths		2 of each
Plastic overalls, eye protection		2 each
Permanent markers, pens		2 each
Stick-on clear label set 1		Blank, Disp. 1, Disp. 2, Disp. 3
Stick-on clear label set 2		3 x (50%, 25%, 12%, 6%),
		Disp. 1, Disp. 2, Disp. 3
Test tube rack (collapsible)		1
Blutac, white card for photographs		1 of each
Plastic teaspoons (stirrers)		6
1ml plastic syringes		4 minimum, 10 optimal
2ml plastic syringes		4 minimum, 10 optimal
5ml syringes		3 minimum, 10 optimal
10ml syringes		6 minimum, 10 optimal
300ml container with screw cap	1	
(for oil sample)		
50ml container with screw caps (for dispersants)	3	
Thermometer (alcohol)	1	
15ml vials	16	
25ml measuring cylinders	2	
100ml beakers	3	
250ml beakers	1	
250ml conical flask with cap	2	
125ml separating funnel with cap	4	
Dispersants	3	
MSDSs for dispersants	3	
~ ~ ~ ~ ~ ~ ~ ~ ~ ~ ~ ~ ~ ~ ~ ~ ~		

### **APPENDIX 2: LIST OF NATIONAL PLAN DISPERSANTS**

# Australian Maritime Safety Authority **Australian Government**

# National Plan Dispersant Type, Suitability, Application and (Summary of data from Material Safety Data Sheets and Manufacturer Information) **Constituent Guide**

Product Name	Manufacturer	Type	Appln Ratio	Use on oils?:	Deliverable from:	In Stockpile:
Ardrox 6120	Chemetall (Australasia) Pty Ltd, Glendenning NSW	II/III, Water-dilutable concentrate¹	1.25	Light - possible Medium – yes Heavy² – yes/possible	Boat (undiluted) – yes Water induction – yes Small Aircraft – yes	AMSA - National Plan
Corexit 9500	Nalco/Exxon Chemicals, L.P.	III, Hydrocarbon Based	1:10 – 1:50	Light - yes Medium – yes Heavy - yes/possible	Boat (undiluted) – yes Water induction – yes Small Aircraft – yes	AMSA - National Plan
Corexit 9527	Nalco/Exxon Chemicals, L.P.	III, Water-dilutable concentrate, non-ionic surfactant <sup>3</sup>	1:20 – 1:30	Light - possible Medium – yes Heavy - yes/possible	Boat (undiluted) – poss. Water induction – yes Small Aircraft – yes	AASA - National Plan AMOSC - AMOSPlan
Dasic Slickgone LTSW	Dasic International Ltd Hampshire. UK.	II/III, Hydrocarbon based concentrate	1:30	Light - possible Medium – yes Heavy - yes/possible	Boat (undiluted) – yes Water induction – yes Small Aircraft – yes	AMSA - National Plan
Dasic Slickgone NS	Dasic International Ltd Hampshire. UK.	III, Water-dilutable concentrate, anionic surfactant <sup>4</sup>	1.25	Light - possible Medium – yes Heavy - yes/possible	Boat (undiluted) – yes Water induction – yes Small Aircraft – yes	AMSA - National Plan AMOSC - AMOSPlan
Shell VDC	Shell	II/III, Hydrocarbon based concentrate <sup>5</sup>	1:30	Light - possible Medium – yes Heavy - yes/possible	Boat (undiluted) – yes Water induction – yes Small Aircraft – yes	AMSA - National Plan AMOSC - AMOSPlan
Tergo R-40	Chemetall (Australasia) Pty Ltd, Glendenning NSW	III, Water-based concentrate, non-ionic surfactant <sup>e</sup>	1:20	Light - possible Medium – yes Heavy - yes/possible	Boat (undiluted) – yes Water induction – yes Small Aircraft – yes	AMSA - National Plan
Tergo 2-005	Chemetall (Australasia) Pty Ltd, Glendenning NSW	III, Water-dilutable concentrate	1:20	Light - possible Medium – yes Heavy - yes/possible	Boat (undiluted) – yes Water induction – yes Small Aircraft – yes	No
Corexit 9550	Nalco/Exxon Chemicals, L.P.	III, Hydrocarbon-based <sup>7</sup>	1:20	Light - possible Medium – yes Heavy - yes/possible	Boat (undiluted) – poss Water induction – no Small Aircraft – yes	No
Shell VDC Plus	Shell	III, Water-dilutable concentrate, mixed anionic and non-ionic	1.25	Light - possible Medium — yes Heaw - wes/nossible	Boat (undiluted) – yes Water induction – yes Small Aircraft – yes	O <mark>N</mark>

<sup>111-76-2)</sup> ACGIH TWA of 25 ppm (120 mg/m³) for skin. STEL 75 ppm (360mg/m³) for skin. STEL 75 ppm (3 For Heavy oils, answers are presented as first / second, based on being applied on - (Heavy oil - good spreading) / (Heavy oil - poor spreading)

AMSA 231 (4/12)

<sup>&</sup>lt;sup>3</sup> Mixture of surfactant esters in mixed oxygenated solvents (& EGMBE at 38%v/v) (2-butoxyethanol present-CAS 111-76-20)

<sup>&</sup>lt;sup>5</sup>EGMBE & biodegradable anionic and non ionic surfactants (10-60%)

Floyethylene Glycol Esters of Oleic and Coconut Fatty acids (major active agent surfactant) TLV >3000pm Acute Oral rat LD >5000mg/kg Solvent = Dipropylene glycol monomethyl ether (10%)