MOVING TOWARDS SUBSTANCE-BASED TOXICITY TESTING TO MEET NEW OSPAR REQUIREMENTS.

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1 ABSTRACT

At the Oilfield Industries Committee (OIC) meeting in February 2002, an Agreement was made by OSPAR Contracting Parties to move from conducting aquatic toxicity testing on offshore chemicals at the preparation level to a substance-based approach. The requirement will be introduced over a period from 2004 to 2007.

This paper will describe the impacts of this Agreement upon chemical suppliers and how the transition process is being managed. First, the magnitude of the impact was evaluated by surveying EOSCA members on the amount of substance-based toxicity data already available. Discussions were held with regulatory agencies to try to rationalise the testing requirements so that unnecessary testing might be avoided. The ability to use freshwater toxicity data was considered as a legitimate alternative to the mandatory marine data. This would align with the European Union White Paper on Chemicals which will also require substance based toxicity testing using fresh water species on all chemicals used within the EU. In addition, a project was initiated by EOSCA to provide a system whereby members could share the cost of substance toxicity testing and also trade existing data. In conclusion, the paper will review the pros and cons of this Agreement for chemical suppliers.

2 INTRODUCTION – OSPAR FRAMEWORK

The OSPAR Commission adopted Decision 2000/2 on a Harmonised Mandatory Control System (HMCS) for the Use and Reduction of the Discharge of Offshore Chemicals[1] in June 2000. The aim of this legislation is to establish a consistent framework within which the amount and harmfulness of chemicals that are discharged in the course of offshore oil and gas exploration and production processes can be reduced. Such chemicals include those used for drilling, production, cementing, completions and workover operations.

The common framework outlined in OSPAR Decision 2000/2 is supported by a number of Recommendations that describe how the Mandatory Control System will work in practice. This framework summarised in a previous paper presented at "Chemicals in the Oil Industry VII"[2]. Under the HMCS, a chemical developed for use on an offshore

installation must pass a Pre-screening Scheme[3] and an assessment of its Hazard Quotient (HQ) before it is permitted to be used offshore and discharged into the sea. The HQ represents the ratio of the Predicted Environmental Concentration (PEC): Predicted No Effect Concentration (PNEC).

To be able to calculate the PNEC and therefore the HQ, toxicity tests are required to be conducted on specified marine species as shown in table 1. These species were selected, not only to represent different physical positions within the marine environment (i.e. water surface, water column and seabed), but also representing links in the food chain i.e. fish feed on crustaceans which feed on algae. Toxicity data is presented to the authority making the assessment on a standard form known as the Harmonised Offshore Chemical Notification Format, or HOCNF, which is described in Recommendation 2000/5[4].

Table 1 *Toxicity Tests required under the HMCS*

Test Required	Test protocol
Algae	72hr EC_{50}: *Skeletonema costatum* ISO/DIS 10253
Crustacean	48 hr LC_{50}: *Acartia tonsa* ISO TC147/SC5/WG2
Fish	96hr LC_{50}: *Schophthalamus maximus*, juvenile OECD 203 modified for marine species
Crustacean – sediment reworker	10 day LC_{50}: *Corophium volutator* PARCOM

Up until OIC in London in March 2003, the toxicity testing had primarily been done at the preparation level (unless the constituent substances were all on the PLONOR list). This was due to it being an absolute requirement for testing to be done on the formulated product by the UK authorities and (although not preferred) an acceptance of this data by other OSPAR countries. The UK preferred toxicity testing to be done on the formulated product, as this assesses the effect of combining chemicals together which could lead to either synergistic or antagonistic effects.

In the HOCNF marine toxicity data are required for all preparations or their constituent substances (unless they appear on the PLONOR list). However, for many offshore chemicals, only data on the toxicity of the preparation are available, and not on the individual component substances. OSPAR Recommendation 2000/4[3] allowed the use of the toxicity data of the preparation to estimate the toxicity of a substance contained in it, taking into account the concentration of the substance in the preparation.

The CHARM User Guide[5] describes how the toxicity data of substances, if available, and that of preparations is used to calculate the HQ of the substances also the preparations containing constituent substances. If both data for PEC and PNEC are available at the substance level: then

$$HQ_{\text{substance i}} = \left(\frac{PEC_{\text{substance i}}}{PNEC_{\text{substance i}}} \right)$$

And

$$HQ_{preparation} = \text{Maximum}\left[\frac{PEC_{substance\ i}}{PNEC_{substance\ i}}\right]_{substance\ i\ to\ n}$$

If data for PEC is available on substance level and data for PNEC is only available on preparation level : then

$$HQ_{preparation} = \text{Maximum}\left[\frac{PEC_{substance\ i}}{PNEC_{preparation}}\right]_{substance\ i\ to\ n}$$

However, the pre-screening assessment requires knowledge of the toxicity of the individual substances. The problem is that for most offshore chemicals, only data on the toxicity of the preparation is available, and little toxicity data exists for the individual component substances. In order to carry out the pre-screening, the Danish Environmental Protection Agency proposed to OIC 2002 the use of a calculation method along the lines of the precautionary approach where substance toxicity test data was not available. Three methods of calculating substance toxicity from preparation data were suggested:

Method I:
When no information on toxicity of the individual substances is available, any one of these could be solely responsible for the measured toxicity. Thus, a conservative approach would be to allocate all of the toxicity of the preparation to each of the substances in the preparation. This would give an estimate of the maximum toxicity of each of the substances:

$$LC50_x = C_x \cdot LC50_{preparation}$$

Method II:
Another approach would be to assume that all of the substances are equally toxic and, thus, as toxic as the preparation, i.e.:

$$LC50_x = LC50_{preparation}$$

This corresponds to equation 31 in the CHARM model. Furthermore, this is the same as allocating the toxicity of the preparation to the individual substances in proportion to the content in the preparation. On average the toxicity would be estimated too high for half of the substances and too low for the other half. It is not possible to identify those substances for which the toxicity is underestimated.

Method III:
An approach that is somewhere in between method I and II is to equally distribute the toxicity of the preparation to each of the substances. This could be considered a “pseudo-conservative” approach, where the toxicity would be overestimated for most of the substances in the preparation. In practice, the toxicity of the preparation is divided by the number of substances (n) contained in the preparation, i.e.:

$$LC50_x = \frac{LC50_{preparation}}{n}$$

EOSCA examined the sensitivity of the 3 methods presented and found that none of the methods were scientifically sound. All of the 3 methods could lead to either overestimated or underestimated results. Method I was favoured by the authorities. However, where substances were present in low concentrations, this method would generally lead to false positive outcomes of high toxicity and would unnecessarily penalise producers of the preparations with a "Substitute" outcome from the pre-screening scheme. . This is shown in Table 2 which shows the calculated toxicity by the three methods for five binary mixtures. The component in the lower concentration is automatically penalised especially by method I, irrespective of its toxicity being substantially lower than higher concentration component, if the toxicity of the higher concentration component is numerically equal to the percentage of that component in the product.

Table 2 *Calculated toxicity for substances at various concentrations in a preparation of calculated toxicity 100 mg/l*

	Content	LC50	Method I	Method II	Method III
		[mg/L]	LC50 [mg/L]	LC50 [mg/L]	LC50 [mg/L]
Preparation A	**100%**	**100**			
Substance Ac	1%	990000	1	100	50
Substance Ad	99%	99	99	100	50
Preparation B	**100%**	**100**			
Substance Bc	10%	900000	10	100	50
Substance Bd	90%	90	90	100	50
Preparation C	**100%**	**100**			
Substance Cc	20%	800000	20	100	50
Substance Cd	80%	80	80	100	50
Preparation D	**100%**	**100**			
Substance Dc	30%	700000	30	100	50
Substance Dd	70%	70	70	100	50
Preparation E	**100%**	**100**			
Substance Ec	49%	500000	49	100	50
Substance Ed	50%	50	50	100	50

3 OTHER DRIVERS

The issue was complicated by two other considerations. First, the European Commission had published a "White Paper"[6] on chemicals which requires the future testing for toxicity (including aquatic toxicity) at the substance level all chemicals being produced and/ or distributed or used within the European Union. The priority is scheduled on the basis of the amounts of substances used. The main aspects of this are as follows.

Both new and existing chemicals marketed in volumes of 1 tonnes (instead of the current 10 tonnes) will need to be registered. For this, manufacturers and importers will need test

their products, carry out a preliminary risk assessment and make proposals for risk management.

Existing substances that lead to high exposure (>1000 tonnes) or cause concern by their known or suspected properties - physical, chemical, toxicological or ecotoxicological – will need to be registered by 2005 (>100 tonnes by 2008 and >1 tonnes by 2012). The risk assessment as such shall still be carried out by the authorities, who might require manufacturers to carry out further specific testing. In the face of the workload, authorities shall focus on areas of major concern. Principally, manufacturers and importers as well as users shall also have responsibility for performing adequate risk assessments.

Substances produced in volumes >1 000 tonnes shall be assessed by 2010 and substances above 100 tonnes by 2012.

Should a manufacturer of a given substance delay the filing of information or test results (objective criteria for what would be a delay are missing), the authority would be entitled to conclude the assessment. It would then pass the dossier to the Commission with a recommendation to apply the precautionary principle and to proceed to risk management measures to the possible extent of a total ban.

As can be seen the testing aspects of these requirements are not proposed to start to come into effect until 2010 and would be phased in over a number of years. Many of the substances used in the oilfield sector are not produced in great quantities and testing of these may not be required until 2012 The testing would be carried out on fresh water species although there appeared to be some provision for using data derived from testing of marine species. However at the time of OIC 2002 these provisions had not been finalised.

There was already concern within the wider chemical industry that the amount of testing that the EU proposals would require would not be physically possible, considering the number of substances that would need to be tested and the number of testing laboratories within the EU. Clearly a requirement of OSPAR to require substance testing on marine species potentially before the EU requirements would only aggravate this situation regarding testing and also place a considerable extra burden on the producers and/or suppliers of offshore chemicals.

In addition to considerations about the White Paper, the issue of substance testing was also been exacerbated by new and imminent legislation in the Netherlands. New regulations would effectively bring forward the EU requirements under the white paper from 2010 to the present. From 2003, any new products put forward for use within the Dutch sector will need to be tested at the substance level. Existing products, which may not have been fully tested and are lacking data such as the fish test will be able to submit this data either on the preparation or on the substances within the preparation.

On consideration of the problems with conducting the pre-screening assessment, the EU White Paper and the imminent introduction of legislation requiring substance testing in the Netherlands, OIC 2002 drew up an Agreement to move from conducting aquatic toxicity tests on offshore chemicals at the preparation level to a substance-based approach. The Agreement stated that for new products, toxicity testing at the substance level will be required from 1 January 2004 and by 1 January 2007 substance toxicity data should be available for all existing products.

4 MAGNITUDE OF POTENTIAL TESTING

To get an idea of the potential impact on the oilfield chemical supply industry EOSCA carried out a survey of its members which number nearly forty companies involved in the manufacture and marketing of chemicals for the oil industry. A questionnaire was produced asking for Members to supply data on the number of products within their range and the number of substances contained within these formulations. The Companies were also asked to give an indication of the toxicity data that they had for the products and the substances and whether this was for marine or freshwater species.

The returns from the questionnaire indicated that many companies have relatively few products with the total number of substances in them being numerically not much more. However some of the drilling mud and production chemicals service companies have substance portfolios running to hundreds. Tables 3 and 4 show the returns from a drilling fluids supplier and a production chemicals supplier with such diverse product ranges.

Table 3 *Drilling Chemical Preparations and Substances with/ without toxicity data*

	Denmark	Norway	Netherlands	UK
Commercial Products registered for use by one Drilling Fluids Supplier.	148	148	?	121
Substances contained within these Products.	248	300	?	240
Substances PLONOR listed.	130	150	?	120
Substances with **full marine** toxicity data.	5	5	5	
Substances with **partial marine** toxicity data.	0	0	0	0
Substances with **full freshwater** toxicity data.	0	0	0	
Substances with **partial freshwater** toxicity data.	0	0		0
Substances with **no** toxicity data.	243	295		235

Table 4 *Production Chemical Preparations and Substances with/ without toxicity data*

	DK	No	NL	UK	OSPAR Region
Commercial Products registered for use by one Production Chemicals supplier.	48	80	10	150	200
Substances contained within these Products.					400
Substances PLONOR listed.					5
Substances with **full marine** toxicity data.					5
Substances with **partial marine** toxicity data.					20
Substances with **full freshwater** toxicity data.					0
Substances with **partial freshwater** toxicity data.					0
Substances with **no** toxicity data?					300

The returns show some interesting features. Notably drilling fluids suppliers generally have more PLONOR listed products in their range than those of production chemical suppliers. Most companies submitting returns had very little or no toxicity data at the substance level. Also the concession to be able to use freshwater data instead of marine data appears to be of limited usefulness at this stage. This may change with time if end point suppliers contact the intermediary material suppliers with a view to asking what freshwater data may be available which they may not have asked for before.

Clearly getting toxicity data on all these substances within the allotted time frame could be difficult from both an economic as well as a logistical point of view. An indication of the costs of carrying out toxicity tests, on the various species to satisfy the requirements of the HMCS, was given in a previous paper[2]. With costs amounting up to £3,600 per substance tested, and with a requirement to test up to 300 substances a company could face a potential total testing cost close to £1 million. With some tests taking several weeks to perform test houses will also be under severe pressure to be able to get all the tests carried out before the required deadline. Given the nature of the business and the types of products supplied by companies in competition with each other, there is definitely potential for different companies to be using some of the same substances making the sharing of existing data or of new data acquisition worth considering. This will be considered later.

5 RATIONALISING TESTING

A feature of the HMCS was that the fish test became mandatory once OSPAR Decision 2000/2 became effective. It soon became clear however that in some countries, laws regarding ethical testing precludes the deliberate killing of fish, as vertebrae, within the protocol of the test, when the toxicity of the material to other species ie algae or crustaceans indicated that this is the most likely outcome. Straightaway the mandatory

nature of testing became "open to interpretation". With a view to again trying to standardise this feature the UK delegation to OIC 2003 introduced a paper which qualified under what circumstances, fish tests would or would not be carried out.

The acceptability of toxicity data carried out on freshwater species in place of that carried out on marine species had been first muted at OIC 2002 in Cadiz. The comparability between the two data sets had been alluded to within the EU Technical Guidance Document on Risk Assessment[7]. This topic was further discussed intersessionally and was also introduced in the UK paper to OIC 2003 in London. The use of toxicity data on freshwater species and clarification of circumstances when the fish test might not be required were both accepted at OIC 2003.

EOSCA feels that this approach should be extended further. To this end it intends to put forward to OSPAR a decision tree approach to testing. This Decision Tree is given in figure 1. It incorporates the acceptability of toxicity data freshwater species and also the use of read across data. This latter aspect needs to be clarified as indiscriminate use of data for read across purposes is not widely accepted. Where we feel it could be used is where data may be available say on a sodium salt of a substance but not for say a potassium salt used in a preparation. It may also be acceptable where data is available for substances containing alkyl chains of one length say C_7 but not of near similar length say C6 or C8 used in a preparation. This might be accepted especially where the toxicity data for a whole homologous series of similar substances is known and show no significant differences throughout the series. This could arise when the toxicity is due to a particular functional group at the end of the chain or elsewhere within the molecule.

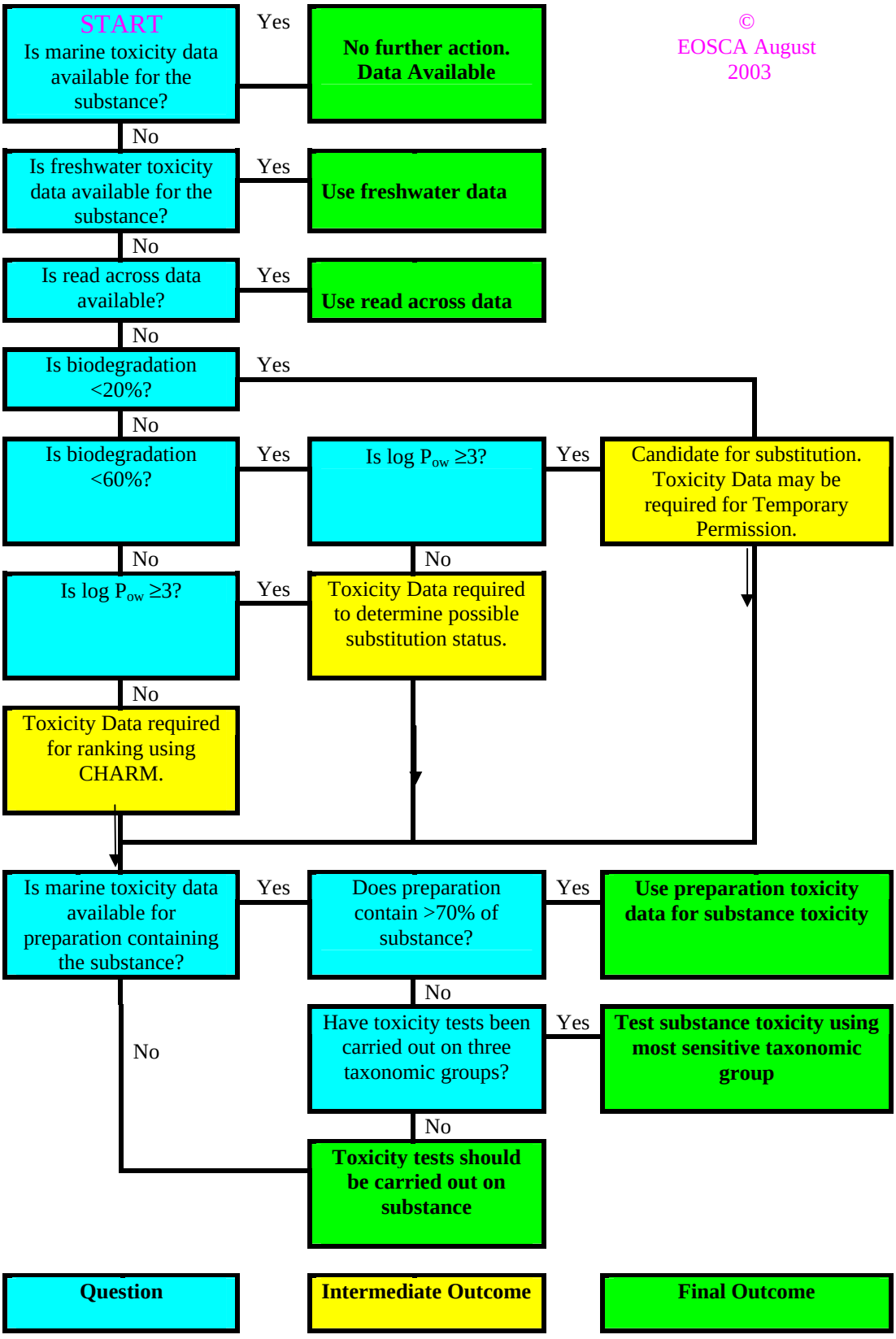

Fig. 1 *Decision Tree to Determine Additional Toxicity Testing*

Another feature of the decision tree is the suggestion that the toxicity of a substance present at more than say 70% in a two component mixture whose toxicity is known might be taken as the same as that of the mixture. This will in fact be lower than actual where the lesser component of the two is more toxic than the higher percentage component. Where the higher percentage component is the most toxic of the two, then this will drive the toxicity of the mixture down and the smaller percentage of a less toxic component will not substantially raise that of the more toxic component.

The decision tree also suggests that if toxicity has been determined on three taxonomic groups for a preparation, then toxicity of the substances within the preparation might only be tested on the taxonomic group showing the most sensitivity in the preparation. This could save a considerable amount of testing for not much more significant data. This approach would automatically introduce penalties in the form of higher assessment factors due to reduced number of species tested. Where HQ's are particularly low these penalties may be of no real consequence.

Whilst the use of the aspects of the decision tree may not be universally accepted, we believe that it may fulfil a role in reducing the testing burden that would fall upon the vendor of the chemical to the oil industry especially in the short to medium term. In the longer term the manufacturer of the substance would need to provide all the data required for assessment under OSPAR rules, under its duty to supply this information to users by virtue of the EU Directive.

6 DATA SHARING SCHEME

It is clear that in the medium term up to 2010 when the EU White Paper requirements are expected to come into play that a substantial amount of new testing of substances will be required. Contracting Parties to OSPAR have often suggested to the chemical supply industry that a significant amount of cost saving may be obtained by sharing the costs of actually getting substances tested between vendors. After all, some substances are used by all the suppliers and it is not necessary perhaps for every user to test every substance. Even if data for a substance is only shared between two Companies then the unit cost for the test is halved. EOSCA investigated the possibilities of doing this quite some time ago but it did not lead to any process. One of the main stumbling blocks was that confidentiality of product formulation between vendors.

EOSCA has more recently revisited the possibility of data and cost sharing and have started a process whereby data for non contentious substances such as solvents etc may be shared. Confidentiality is being achieved by virtue of the fact that the "go between" between companies is the EOSCA Secretary who is independent of the Member Companies. Previously this role was fulfilled on a volunteer basis from the Company Membership.

Companies requesting data or having data to offer to share notify the EOSCA Secretary who then informs Members what is being requested and what is on offer. Requests are made formally by completing a form listing all the data for a particular substance that a Company may need, specifying the test species and the protocols that they would prefer or would be willing to accept. The form is also used if a Company has data it is willing to

share under the scheme. The form also requires species tested and protocols used to be listed. The Secretary checks data requirements against data availability and starts a dialogue with the two parties if there is a straight match or a potential match of data. Prior to data actually being exchanged Members sign a confidentiality agreement with the Secretary. This allows the Secretary access to confidential reports or other documents, which are used to verify that data has actually been acquired for the substances involved. Letters of access to both the data supplying and receiving Companies may be used to inform Regulators that the data has been supplied or received via the scheme. This provides the audit trail necessary should any Regulator need to verify data reports.

Confidentiality is preserved throughout the process even to the point of keeping invoice transactions separate from the day to day running of the Association.

At the time of writing only two companies have shared data on one substance. There is an increasing amount of interest by EOSCA Members to share data and from the small beginning it is hoped that more data will ultimately be exchanged. Where more than one Member requests data for the same substance but no data exists or other Members with the data are unwilling or unable to share, then it is hoped that a collaborative approach to getting substance tested may be possible. The ultimate aim is for all parties to get relevant data at a reduced cost.

7 IMPACT ON THE OFFSHORE CHEMICAL SUPPLY INDUSTRY

As indicated above as and when measures outlined in the EU White Paper come into force, manufacturers in the EU or importers of substances to the EU will have to provide all the datasets needed to fulfil the requirements of the Registration, Evaluation and Authorisation of Chemicals system (REACH). These will automatically fulfil the requirements for OSPAR assessment under HMCS. There is a concern within the oilfield chemical supply industry that where a substance is manufactured outside of the EU and is imported in only relatively small tonnages to satisfy the oilfield market, then the size of that market may not sustain the cost of chemical by the manufacturer. There is a concern that the palette of products available for use in oilfield chemical formulations will reduce and could make some products unavailable without the vendor carrying the cost of all the testing required.

There could be a neutrality of cost impact if other manufacturers are effectively testing substances which might otherwise be tested by the chemical vendors. Only time will tell how this pans out and whether the range and diversity of formulations will decrease.

The introduction of HMCS was supposed to create a degree of harmonisation which should have been very positive for the chemical supply industry. Standardisation of the reporting formats (HOCNF), environmental test protocols, and the use of the pre-screening scheme and CHARM should have helped chemical suppliers to source the required data more efficiently. The transparency of the system should have enabled suppliers to invest resources into products that will be more successful under the scheme i.e. those with good environmental performance.

Practically there are still significant differences between the National schemes which are giving rise to confusion for companies which register products for use in more than one country. Different time scales of Registration mean that new data is required in one

country long before another. Also the risk assessment process is different in all the major oil producing countries.

Before the EU proposals come into effect the onus will still be on the ultimate supplier to the operator customer, to provide data for Registration and Risk Assessment. With the use of substance based data there must be an ultimate harmonisation of process. This will be of benefit to the oilfield chemical supply industry. At least to those that survive the intervening years.

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