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A review of toxicity testing conducted on European refinery effluents in 2010 and 2013

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A review of toxicity testing conducted on European refinery effluents in 2010 and 2013

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ABSTRACT

This report provides an initial assessment of the extent to which toxicity testing is carried on European refinery effluents, and the types of test being used. The analysis is based on responses given to Concawe water use and effluent quality surveys completed for the 2010 and 2013 reporting years.

Toxicity testing was predominantly carried out to fulfil permit requirements, or at the request of regulators. However, the part of effluent streams being voluntarily monitored with toxicity testing increased from 6% in 2010 to 27% in 2013 (from 10% to 27% for refineries reporting in both years). Voluntary toxicity testing was usually performed on an annual basis. The most frequently reported toxicity test in both the 2010 and 2013 surveys was *Daphnia magna*, with toxicity to fish eggs also commonly used in 2013. Both of these tests were usually performed on a quarterly basis. In both years most of the testing was carried out on treated process waters (52% of effluent streams in 2010 and 46% in 2013).

KEYWORDS

Toxicity test, effluent, wastewater, treatment, refinery, survey, *Daphnia magna.*

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SUMMARY

Concawe has been gathering and compiling data on water use and effluent quality for European oil refinery installations since 1969 (Concawe, 2012). The primary focus of these survey questionnaires has been the volume, type, and quality of the water being used and discharged by the industry. However, the 2010 and 2013 surveys also included a new section to collect information on the use of toxicity tests for refinery effluent monitoring.

Refineries from the EU-28 countries + Norway and Switzerland participated in both surveys, with a response rate of 89% in 2010 and 76% in 2013. In the 2010 survey, 37 out of the 100 responding refineries (37%) indicated that at least one of their effluent streams had some form of toxicity test performed, whereas the corresponding number in the 2013 survey was 27 out of 79 responding refineries (34%).

In both survey years, at least one refinery within each of the eight country groups reported using toxicity tests, suggesting that the use of such tests is fairly widespread. Toxicity testing was predominantly carried out to fulfil permit requirements, or at the request of regulators. However, the part of effluent streams being voluntarily monitored with toxicity testing increased from 6% in 2010 to 27% in 2013 (from 10% to 27% for refineries reporting in both years). Voluntary toxicity testing was usually performed on an annual basis.

The most frequently reported toxicity test in both the 2010 and 2013 surveys was *Daphnia magna*, with toxicity to fish eggs also commonly used in 2013. Both of these tests were usually performed on a quarterly basis. In both years most of the testing was carried out on treated process waters (52% of effluent streams in 2010 and 46% in 2013).

The findings from this preliminary assessment suggest that further insights could be gained by optimising the design of future water/ effluent questionnaires.

1. INTRODUCTION

Concawe has been gathering and compiling data on water usage and effluents from European oil refinery installations since 1969. In recent years, data gathering on water usage and effluents has utilized questionnaire surveys in MS Excel format submitted to member installations on a triennial reporting interval. The primary focus of these questionnaires has been on the volume, types, and quality of the water being used and discharged by the industry. However, the surveys for the 2010 and 2013 reporting years also included new adaptations which provide further detail on the utilization of toxicity testing on effluent streams within the industry. These additional questions asked responders to:

1) indicate if toxicity testing was conducted on the given effluent stream;

2) indicate why the toxicity test was performed;

3) indicate which toxicity test(s) were being conducted.

This report provides a summary of the industry utilization of toxicity testing of refinery effluent streams based on the 2010 and 2013 questionnaire responses.

2. RESPONSE TO QUESTIONNAIRE

The questionnaire was distributed to Concawe member companies in 2011 and 2014 for 2010 and 2013 operational data, respectively. In 2013, a total of 79 responses of 104 potential respondents[1] (76% response rate) were collected from refineries that represent a wide geographic scope and range of refinery types/complexities. In comparison, 100 refineries out of a potential of 112 responded to the 2010 survey (89% response rate). The lower number of potential respondents in 2013 reflects the number of refineries that closed in this time period.

In the 2013 survey, 27 out of 79 responding refineries (34%) indicated that at least one of their effluent streams had some form of toxicity test performed. A total of 52 effluent streams across the industry were subjected to some form of toxicity testing. This accounts for 32% of the total number of effluent streams indicated in the 2013 survey responses and 17% of the total effluent volume having some form of toxicity test performed.

In the 2010 survey, 37 out of the 100 responding refineries (37%) indicated that at least one of their effluent streams had some form of toxicity test performed. A total of 63 effluent streams across the industry were subjected to some form of toxicity testing. This accounts for 29% of the total number of effluent streams indicated in the 2010 survey responses and 18% of the total effluent volume having some form of toxicity test performed.

In both survey years, at least one refinery within each of the eight country groups (**Figure 1**) indicated some form of toxicity testing. This indicates adoption of toxicity testing across the industry as a whole. As shown in **Figure 2**, in 2010 over 50% of the responding refineries in France and Germany registered at least one effluent stream that performed toxicity testing. In 2013 over 50% of the refineries in the Mediterranean countries group registered at least one effluent stream that performed toxicity testing. It is also interesting to note that the relative percentage of responding refineries conducting toxicity testing in France reduced from 73% in 2010 to 25% in 2013, whereas the relative percentage of refineries in the Mediterranean and UK & Ireland increased between 2010 and 2013. Additional information would need to be collected to determine the reason for all these apparent shifts.

[1] The number of potential respondents represent the number of refineries within the EU-28 countries plus Norway and Switzerland that were declared to be operational in 2013

Figure 1: Geographic Extent of Country Groupings

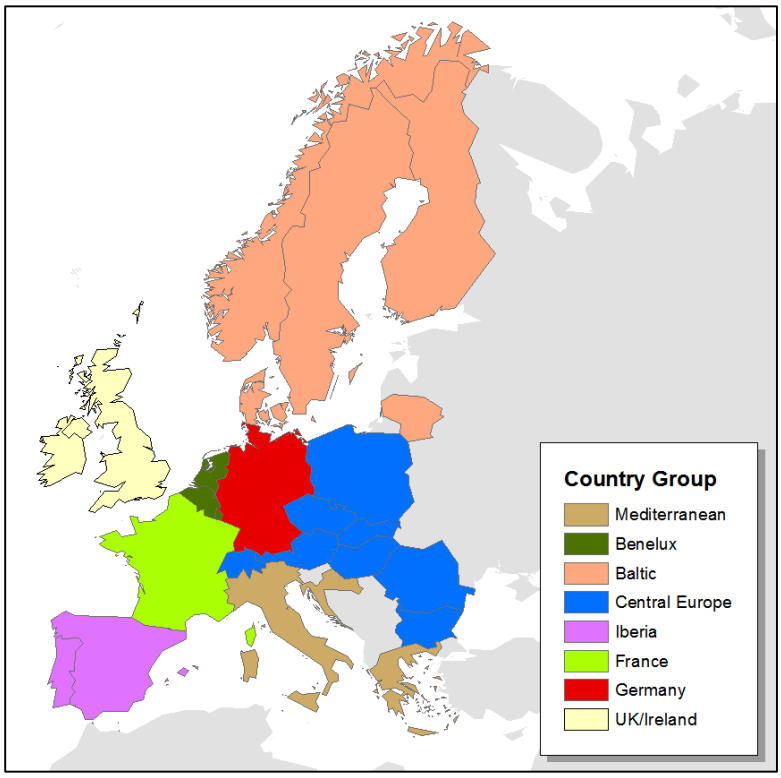
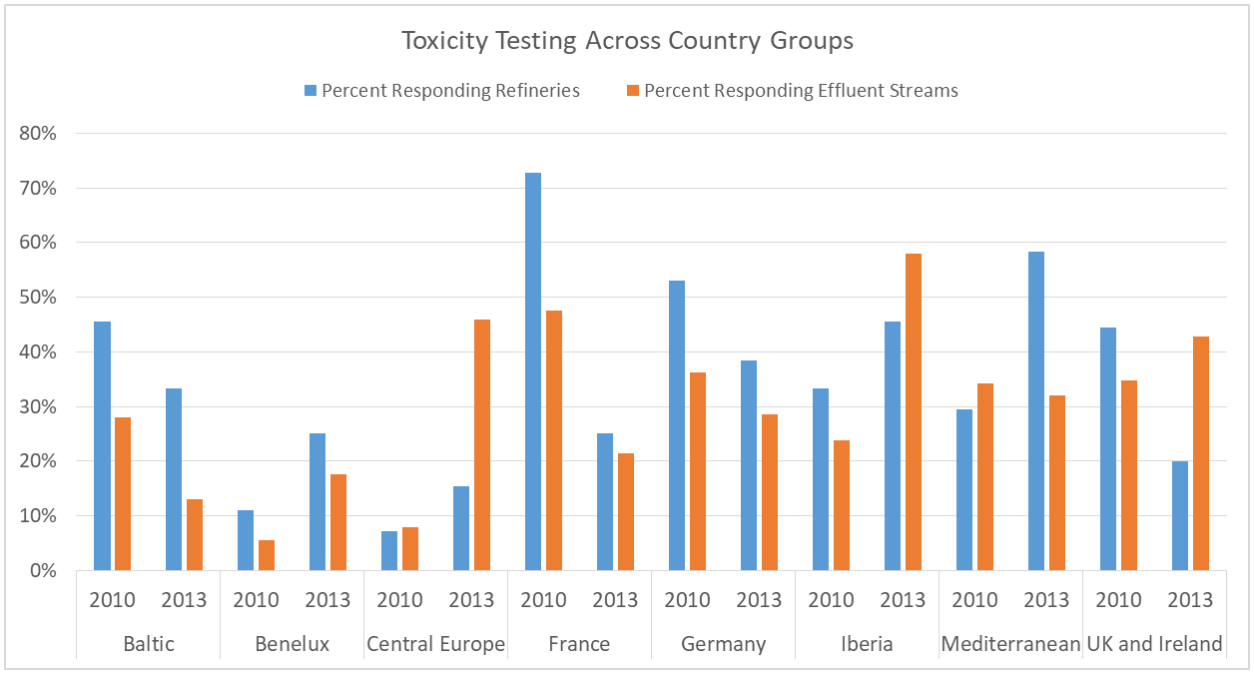


Figure 2: Percent of responding refineries and their effluent streams that indicated having some form of toxicity test performed for each country group.



2.1. REASON FOR PERFORMING TOXICITY TESTS

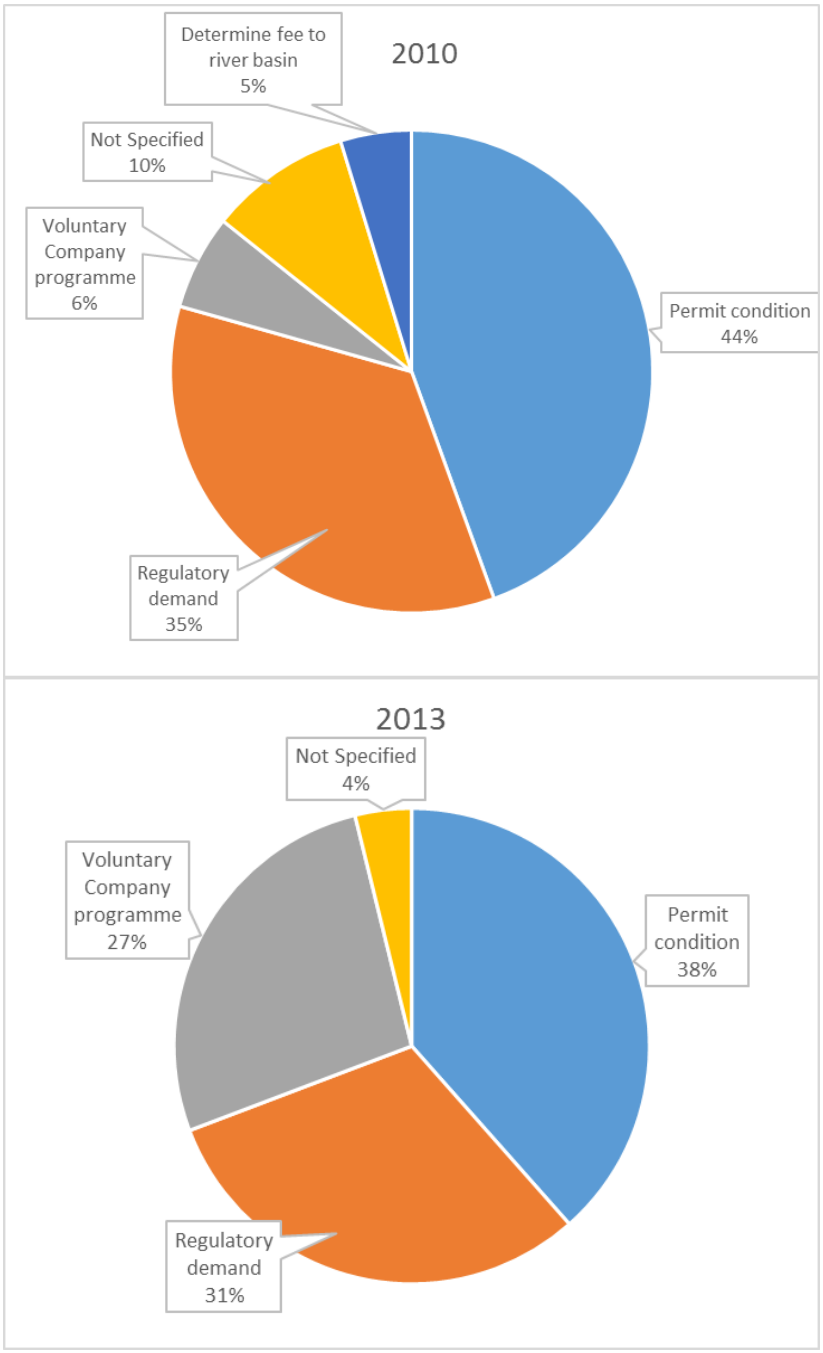
In both survey years, the predominant reason listed for performing toxicity testing was to satisfy permit conditions, followed very closely with regulatory demand. **Table 1** provides a list of reasons given for performing the toxicity testing, whereas **Figure 3** provides a pie chart showing the relative percentages of unique effluent streams being monitored for each reason.

Table 1: Reason indicated for performing toxicity testing

Reason for Toxicity Testing	Number of Effluent Streams in 2010 data	Number of Effluent Streams in 2013 data
Permit condition	28	20
Regulatory demand	22	16
Not Specified	6	2
Voluntary Company programme	4	14
Determine fee to river basin	3	N/A

Interestingly, as shown in **Table 1** and **Figure 3**, there were a number of refineries from each survey year that voluntarily performed the toxicity tests. The relative percentage of effluent streams voluntarily monitored with toxicity testing increased from 6% in 2010 to 27% in 2013. When limiting the analysis to only those refineries that responded in both 2010 and 2013, the increase in voluntary toxicity testing still increased from 10% in 2010 to 27% in 2013. This indicates that the increase is a measured change and not an artefact of which refineries were included in the datasets. More information would need to be gathered to determine the reason for the increase in voluntary adoption of toxicity testing.

Figure 3: Relative percentage of the reason effluent streams were monitored with toxicity testing.



2.2. TYPE OF TESTS PERFORMED

In both survey years the majority of refineries which indicated conducting a toxicity test did not provide a specific test type. This is most likely due to the way the question was phrased in the questionnaire[2]. Many users assumed the question was referring to general tests performed on the effluent stream water rather than specifically the toxicity tests performed. As a result, there are many cases where users indicate there were toxicity tests performed but then fail to specify those tests.

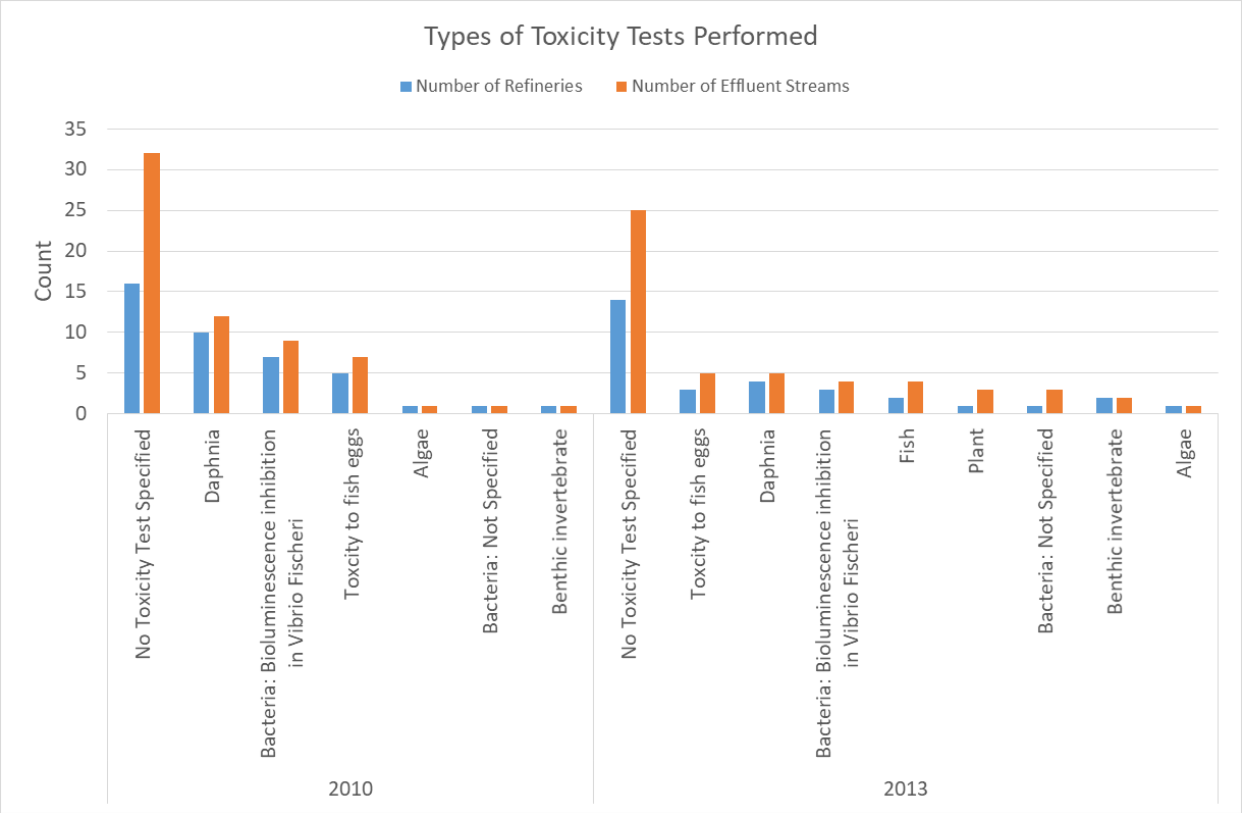
In addition, there were three effluent streams that only provided generic descriptions of the toxicity tests performed. Descriptions such as "Toxicity tests on 2 marine organisms" are not readily categorized and therefore for the purposes of these analyses, they were included in the "No Toxicity Test Specified" category. **Figure 4** provides a summary of the various toxicity tests performed along with their relative counts of refineries and effluent streams conducting each test.

[2] For each effluent stream the questionnaire asked three questions related to toxicity testing (out of 13 total effluent-specific questions):

(1) "Is this effluent tested prior to final discharge to determine their toxicity to aquatic organisms (fish, invertebrates, algae or microorganisms)?"
(2) "Why are these aquatic toxicity tests performed?"
(3) "What tests are carried out? (Please provide a narrative on tests performed)?"

The last question was deemed unclear since many responders added general testing performed on the effluent stream, i.e. pH, Biological Oxygen Demand (BOD), Chemical Oxygen Demand (COD), Total Suspended Solids (TSS), etc., instead of the requested specifics of the toxicity tests being performed.

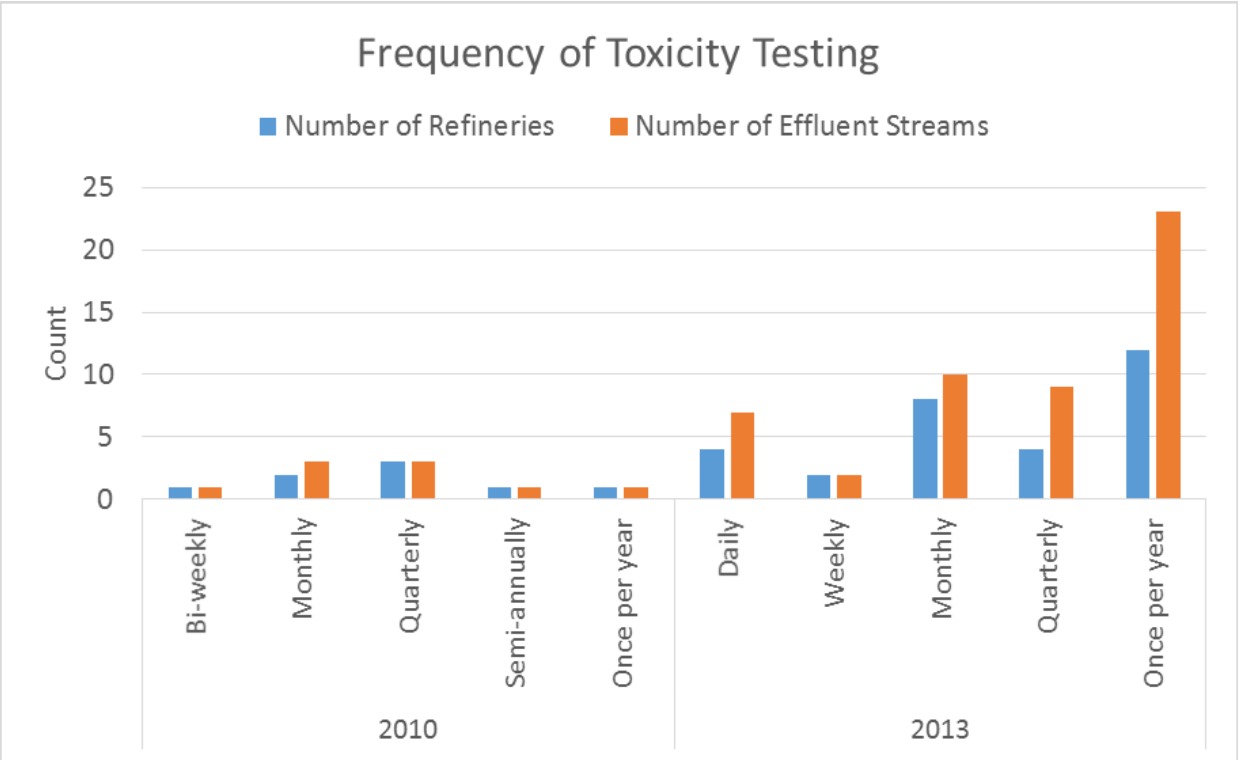
Figure 4: The number of refineries and effluent streams which conducted the various types of toxicity testing.



Of the refineries that did specify a toxicity test, the predominant test conducted in 2010 was *Daphnia magna* (41% of effluent streams) and in 2013 it was *Daphnia magna* and Toxicity to fish eggs (25% of effluent streams each). It is important to note that in the 2013 survey, four effluent streams from two refineries indicated a general toxicity test described as "fish". It is unclear if these tests were conducted on fish eggs as many of the other refineries indicated or on fish specimens. For this reason, the "fish" test descriptions were treated as a separate toxicity test for these analyses. However, if these toxicity tests were actually on fish eggs then that would be the predominant test type in 2013.

The questionnaire in 2010 did not specifically request the responders to indicate the toxicity testing monitoring frequency being employed for each effluent stream. However, many responders provided monitoring frequency information within the notes section which was able to be captured and classified. On the other hand, the 2013 survey requested that responders classify the frequency of toxicity monitoring in one of the following categories: Daily, Weekly, Monthly, Quarterly, and Once per year. **Figure 3** provides a summary of toxicity monitoring frequencies for those effluent streams with data.

Figure 5: Frequency of toxicity testing monitoring



While the 2010 data cannot be considered complete (only 14% of effluent streams had corresponding monitoring frequency information in 2010, while it was 97% in 2013), it appears that effluent streams were most commonly monitored for toxicity on a quarterly basis. In 2013, the most common monitoring frequency was annually, however a large number of effluent streams were also monitored for toxicity on a monthly and/or quarterly basis. As shown in **Table 2**, the majority of effluent streams that are monitored annually are those that conducted toxicity tests on a voluntary basis whereas those conducted on a monthly or quarterly basis were being monitored to meet regulatory demands or fulfil permit conditions.

Table 2: Frequency of toxicity testing and their predominant reason for conducting the tests.

Data Year	Test Frequency	Reason for toxicity testing	Number of Refineries	Number of Effluent Streams
2013	Daily	Permit condition	3	6
		Regulatory demand	1	1
		Voluntary Company programme	0	0
		Not Specified	0	0
	Weekly	Permit condition	0	0
		Regulatory demand	1	1
		Voluntary Company programme	1	1
		Not Specified	0	0
	Monthly	Permit condition	4	6
		Regulatory demand	4	4
		Voluntary Company programme	0	0
		Not Specified	0	0
	Quarterly	Permit condition	2	2
		Regulatory demand	2	5
		Voluntary Company programme	0	0
		Not Specified	2	2
	Once per year	Voluntary Company programme	4	13
		Regulatory demand	4	5
		Permit condition	4	5
		Not Specified	0	0

Notes: Some refineries had two or more effluent streams that were monitored at different frequencies. These refineries are represented more than once in this table. Therefore the total sum of refineries in this table is larger than the total unique number of refineries. One refinery did not include a test frequency for one of its 2013 effluent streams and is not included in this table. However, the refinery had listed 'Permit condition' as the reason for conducting toxicity testing this missing effluent stream.

2.3. TYPES OF EFFLUENTS TESTED FOR TOXICITY

In both survey years, the effluent stream most often tested for toxicity was "process water" (52% in 2010 and 47% in 2013), as shown in **Table 3**. This was the case for each country group as well as the industry as a whole.

Table 3: Effluent stream types that received toxicity testing

Effluent Stream Type	Number of Refineries in 2010 data	Number of Effluent Streams in 2010 data	Number of Refineries in 2013 data	Number of Effluent Streams in 2013 data
Process water (TREATED)	28	33	18	24
Process water (UNTREATED)	1	1	1	1
Cooling water (UNTREATED)	4	6	2	2
Cooling water (TREATED)	2	2	1	3
Storm or rain water (TREATED)	3	4	1	3
Storm or rain water (UNTREATED)	2	2	2	3
Domestic effluent (TREATED)	1	1	0	0
Desalination Process Water	1	1	0	0
Groundwater remediation	1	2	1	2
Mixture	3	3	0	0
Mixture: Process and Storm water	2	2	2	2
Mixture: Process and Cooling water	0	0	2	3
Mixture: Process, storm and domestic water	1	1	1	1
Mixture: Cooling and Storm water	2	2	1	1
Other	2	2	4	5
Not Specified	1	1	2	2
Total	**54[1]**	**63**	**38**	**52**

[1]The total number of refineries is not the same in **Table 3** as in **Table 4** due to one refinery only being counted once in **Table 3** (reporting two effluent streams of the same type) whereas it is counted twice in **Table 4** (the two reported effluent streams do not have the same treatment)

Note: Some refineries had two or more effluent streams that were produced by different effluent stream types. These refineries are represented more than once in this table. Therefore the total sum of refineries in this table is larger than the total unique number of refineries.

In addition, both years' data show that effluent waters which were first treated with a 3-stage wastewater treatment process were considerably more most often tested for toxicity compared to effluents going through a single treatment step or being untreated, as shown in **Table 4**. This coincides with 3-stage wastewater treatment being the most common treatment process utilized by the industry (Concawe, 2012).

Table 4: Effluent stream treatment process for effluent waters that received toxicity testing

Effluent Stream Treatment Process	Number of Refineries in 2010 data	Number of Effluent Streams in 2010 data	Number of Refineries in 2013 data	Number of Effluent Streams in 2013 data
Three stage wastewater treatment incl. biological stage	21	23	24	26
Not treated	8	9	3	3
Chemical	7	9	3	4
Biological	7	7	1	1
Physical/Mechanical	5	7	5	8
Not Specified	5	6	1	2
Send to external facility	2	2	1	1
Total	**55**[1]	**63**	**38**	**45**

[1]The total number of refineries is not the same in **Table 3** as in **Table 4** due to one refinery only being counted once in **Table 3** (reporting two effluent streams of the same type) whereas it is counted twice in **Table 4** (the two reported effluent streams do not have the same treatment)

Note: Some refineries had two or more effluent streams that were each treated by different process. These refineries are represented more than once in this table. Therefore the total sum of refineries in this table is larger than the total unique number of refineries.

3. CONCLUSIONS

Data from the 2010 and 2013 Concawe refinery effluent surveys has been used to assess the use of toxicity tests on effluent streams. In both survey years, at least one refinery within each of the eight country groups reported using toxicity tests, suggesting that the use of such tests is fairly widespread.

Toxicity testing was predominantly carried out to fulfil permit requirements, or at the request of regulators. However, the number of refineries voluntarily conducting toxicity tests increased from 6% in 2010 to 27% in 2013 (from 10% to 27% for refineries reporting in both years). Voluntary toxicity testing was usually performed on an annual basis.

The most frequently reported toxicity test in both the 2010 and 2013 surveys was *Daphnia magna,* with toxicity to fish eggs also commonly used in 2013. Both of these tests were usually performed on a quarterly basis.

In both years most of the testing was carried out on treated process waters (52% of effluent streams in 2010 and 46% in 2013).

The findings from this preliminary investigation suggest that further insights could be gained by optimising the design of any future questionnaires. Improvements could include:

- Providing respondents with a drop-down list of predefined test species to minimize the collection of generic or non- specific records.
- Avoiding that respondents can leave data entry fields blank, that are important for the data analysis.
- Collecting additional details on the toxicity tests being performed, such as exposure method (e.g. EN ISO 6341, etc.), exposure duration, exposure type (e.g. static, renewal, flow-through, etc.), effect studied (e.g. survival, growth, reproduction, etc.), and what test endpoint was used (e.g. half maximal lethal concentration (LC50), half maximal effective concentration (EC50), 10% of maximal effective concentration (EC10), no observed effect concentration (NOEC), lowest observed effect concentration (LOEC), etc.)

Future surveys could also collect toxicity test results to provide insight into environmental performance. However, the interpretation of this data would be difficult unless contemporaneous analytical data was also collected to put the results in context (for example, to rule out confounding factors).

4. GLOSSARY

BOD	Biological oxygen demand
COD	Chemical oxygen demand
EC10	10% of maximal effective concentration
EC50	Half maximal effective concentration
EN ISO 6341	Method for the determination of the acute toxicity to *Daphnia magna* Straus (*Cladocera, Crustacea*)
LC50	Half maximal lethal concentration
LOEC	Lowest observed effect concentration
NOEC	No observed effect concentration
TSS	Total suspended solids

5. REFERENCES

Concawe (2012) Trends in oil discharged with aqueous effluents from oil refineries in Europe – 2010 survey data. Report No. 6/12. Brussels: Concawe

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