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A review of toxicity testing conducted on European refinery effluents in 2010, 2013, 2016, and 2019

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A review of toxicity testing conducted on European refinery effluents in 2010, 2013, 2016, and 2019

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ABSTRACT

This report provides an assessment of the extent to which toxicity testing is carried on European refinery effluents, and the types of tests being used. The analysis is based on responses given to Concawe water use and effluent quality surveys completed for the 2010, 2013, 2016, and 2019 reporting years. The overall response rate of toxicity data has remained similar (~37%) over the period despite a general decrease in both the number of operational refineries and the number of responses to the surveys.

Toxicity testing was predominantly carried out to fulfil permit requirements, or due to regulatory demand. Voluntary monitoring continues to be performed on an annual basis across many country groups. The most frequently reported toxicity tests across the four survey years were *Daphnia magna* and *vibrio fischeri*/Microtox.

KEYWORDS

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

INTERNET

This report is available as an Adobe pdf file on the Concawe website (www.concawe.org).

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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, 2013, 2016, and 2019 surveys also included a 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 all surveys, except for the United Kingdom (UK) and Ireland, who only participated in the 2010 and 2013 surveys. The response rates were 89% in 2010, 76% in 2013, 73% in 2016, and 64% in 2019. Of the responding refineries 37 out of 100 (37%) in 2010, 27 out of 79 (34%) in 2013, 18 out of 76 (24%) in 2016, and 24 out of 65 (37%) in 2019, indicated that at least one of their effluent streams had some form of toxicity test performed.

Excluding the UK and Ireland, at least one refinery within each of the remaining seven country groups reported using toxicity tests for each of the four surveys, suggesting that the use of such tests is widespread. Toxicity testing was predominantly carried out to fulfil permit requirements or to comply with regulatory demands.

The most frequently reported toxicity test in all four surveys was *Daphnia magna* with luminescent bacteria (*vibrio fischeri*) also being commonly used. Fish eggs (*Danio rerio*) were used largely in 2013. Toxicity tests were most commonly conducted annually and monthly in the 2010-2019 surveys.

1. INTRODUCTION

Since 1969, Concawe has been gathering and compiling data on aqueous effluents from European oil refinery installations. Surveys have been completed at 3-5 years intervals with the survey design updated over time following scientific and legislative developments. Since 2010, data collection has included water uses within installations as well as data on toxicity testing. From 2016, a web-based data collection system has been used, which greatly facilitates data gathering and quality assurance. While respondents were required to enter the toxicity section, it was optional to provide the requested information (Concawe 2020).

A review of toxicity testing data reported in 2010 and 2013 was published by Concawe in 2018 (Concawe, 2018). It provided an initial assessment of the extent to which toxicity testing was carried on European refineries' effluents and the types of tests being used. This 2022 report provides a summary of the industry utilization of toxicity testing of refinery effluent streams based on the 2010, 2013, 2016, and 2019 questionnaire responses. It complements and updates the 2018 toxicity review by including reported data from the 2016 and 2019 surveys and by addressing the following analyses:

- The percentage of responding refineries that indicated having some form of toxicity testing conducted in their effluents;
- The reasons indicated for performing the toxicity tests;
- The types of toxicity tests performed and their frequency;
- The types of toxicity tests performed by receiving environment and by country;
- Comparison between the 2010, 2013, 2016 and 2019 survey responses; and
- General conclusions from the review on the most common tests, reasons, frequency, and countries where the tests were performed.

2. RESPONSE TO QUESTIONNAIRE

Over the 2010 to 2019 period, the information requested within the questionnaire has undergone multiple changes, as shown in **Table 1**. These changes were made to provide more accurate and informative data from the operational sites about their toxicity data. For example, in the 2010 and 2013 surveys there was no distinction between effluents and outfalls. Whereas, in 2016 and 2019, members were asked to provide information on the toxicity test(s) performed on effluent streams versus outfalls to create a distinction between the two. Effluents are defined as wastewater streams that receive no further treatment or monitoring prior to discharge (but which may undergo dilution with other wastewater streams prior to the discharge point). An outfall is a physical discharge location and may include one or more different effluent streams depending on how streams are merged into the outfall.

In 2016, members were also asked to provide some additional detailed toxicity information (see **Table 1**). This information was a function of test type and does not seem useful for additional analyses and it was not requested in the 2019 survey. Additionally, in 2016 respondents were not required to enter the toxicity section of the questionnaire, which led to a reduction in toxicity data responses that year. In the 2019 survey the user was required to enter the toxicity section which was structured with picklists and pulldowns so that the data provided would be more uniform for analysis.

Table 1. Metrics Available for Analysis

Toxicity Metrics Available	Survey Year				Comment
	2010	2013	2016	2019	
Test Reason	X*	X*		X *	
Test Frequency	X*	X*	X *	X *	
Test Name/Type/Species Tested	X	X	X	X *	2010/13 derived from user comments, 2016 is a free form field, 2019 is a dropdown
Receiving Water Type	X*	X*	X	X	Picklist 2010/13 Calculated 2016/19
Tox type by Predominant Water Type / Specific Water Uses	X	X	X	X	2010/13 had general effluent water types (i.e., process water or cooling),2016/19 are linked to specific and detailed uses.
Was effluent treated? And Type of Treatment	X	X	X	X	Derived from other metrics
Percentage of responding Sites/Outfalls	X	X	X	X	Calculated
Tox Tests by Country Group	X	X	X	X	Calculated
Tox Tested on Effluent versus Outfall			X	X	Test is linked to either effluent or combined outfall 2016/19. No distinction between effluent/ outfall in 2010/2013
Tox Endpoint			X*		This field information is a function of test type and does not seem useful for additional analyses. Only specifically asked in 2016 survey
Tox Effect Studied			X*		
Tox Exposure Type			X*		
Tox Exposure Duration			X		

** = Drop Down/Pick List*

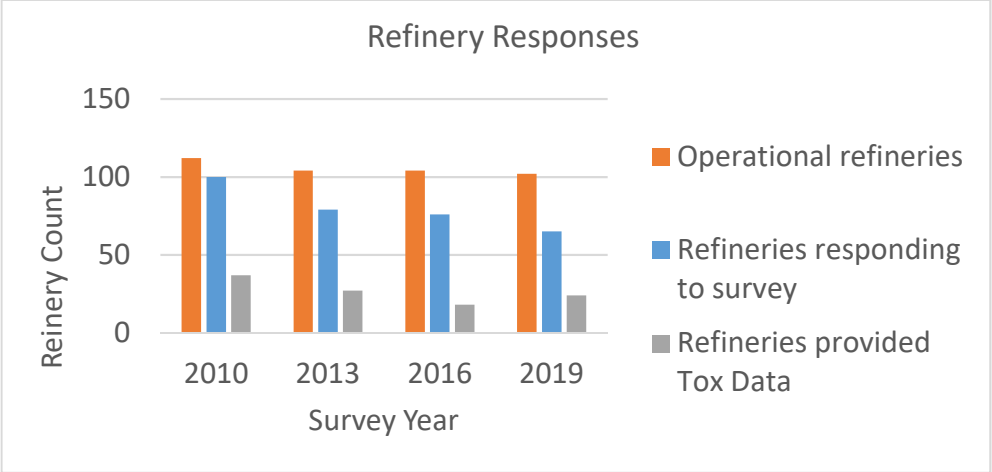
Figure 1 shows the eight country groupings with refineries that reported toxicity data in at least one of the four surveys from 2010 to 2019.

Figure 1. Country Groupings

2.1. RESPONSE RATE

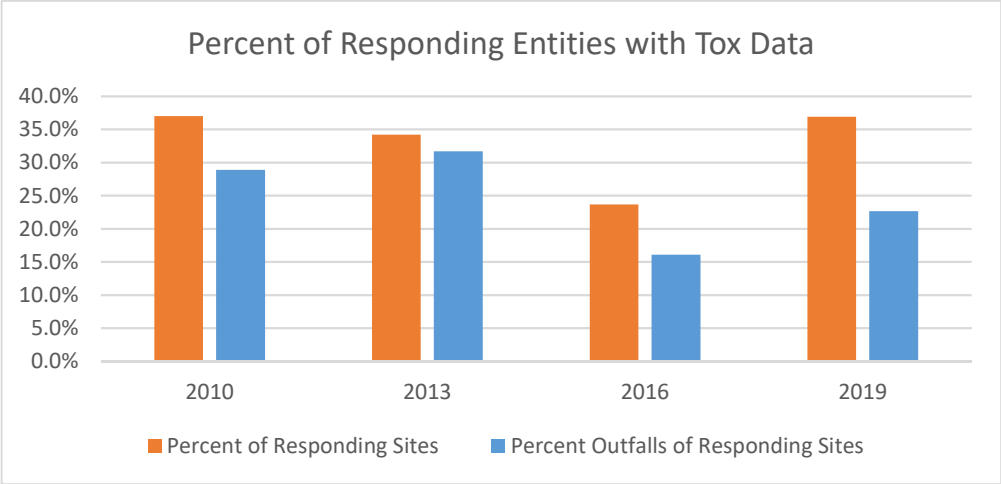
The questionnaire was distributed to Concawe member companies in 2011, 2014, 2017, and 2020 for the 2010, 2013, 2016, and 2019 operational data, representing a wide geographical scope and range of refinery types and complexities. Response rates for 2010 and 2013, i.e., the number of refineries that responded to the survey out of the total number of available (operating) refineries, was 89% for 2010 and 76 % in 2013. In the 2016 survey, 76 out of the 104 open refineries responded the survey (73% response rate). This value reduced in the 2019 survey, with 65 out of the 102 operational refineries (64% response rate) completing the survey. The lower number for the potential respondents reflects the number of refineries that have ceased operation in this time period (**Figure 2**).

Figure 2. Number of Refineries that responded with Toxicity Data



As reported in the 2018 toxicity review report and shown in **Figure 3**, 37% of reporting refineries had some form of toxicity testing in at least one of their effluents in 2010, while in 2013 that rate was 34%. This corresponds to a total of 63 and 52 effluent streams across the industry respectively for 2010 and 2013, with some form of toxicity testing performed. In the 2016 survey, 18 out of the 76 responding refineries (24%) indicated that at least one of their effluent streams had some form of toxicity test performed, with a total of 23 effluent streams across the industry subjected to some form of toxicity testing. This accounts for 16% of the total number of effluent streams indicated in the 2016 response.

Figure 3. Percent of Refineries that responded with Toxicity Data



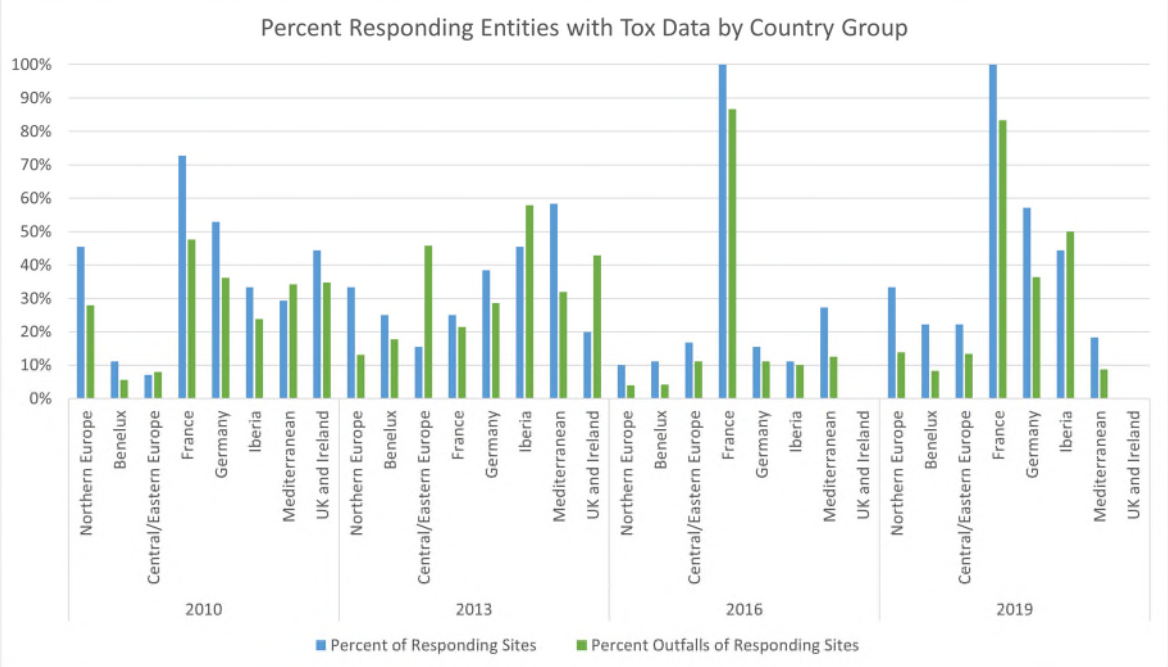
In the 2019 survey, 24 out of the 65 responding refineries (37%) indicated that at least one of their effluent streams had some form of toxicity test performed with a total of 29 effluent streams across the industry subjected to some form of toxicity testing. This accounts for 23% of the total number of effluent streams indicated in the 2019 response.

The lower percentage of toxicity tests reported in 2016 (24%) compared to other years (37% in 2010, 34% in 2013 and 37% in 2019) is likely due to the fact that in 2016 the user was not required to enter the toxicity section as part of the questionnaire.

Except for the United Kingdom and Ireland, in the past two surveys, at least one refinery within each of the remaining 7 country groups (**Figure 1**) indicated some form of toxicity testing. This continues to show the adoption of toxicity testing across the industry (**Figure 4**). In 2016 and 2019, all the responding sites operating in France provided toxicity data for over 80% of the outfalls. This is a rise from the 2013 data where France's response rate dropped from 73% of sites (48% of outfalls) in 2010 to 25% of sites (21% of outfalls) in 2013. In 2019 Iberia and Germany showed similar response rates to those in 2013. Though in general, response rates in 2019 are higher than those in 2016, where users were not required to enter the toxicity section, they are still not as high as in 2010 and 2013.

Figure 4 also shows that there is a greater number of outfalls than available refineries, and that toxicity data is not available for all outfalls. The fact that percent of outfalls with toxicity testing exceeded the percent refineries in 2013 for United Kingdom and Ireland, Central Europe, and Iberia indicates that the toxicity testing was isolated to a smaller number of refineries, but those refineries had multiple outfalls with toxicity testing carried out.

Figure 4. Percentage of Available Refineries and Outfalls with Toxicity Data per Country



When considering the receiving environments of effluents with toxicity testing, **Figure 5** shows that a slightly larger number of toxicity tests were carried out on effluents discharged into salt/brackish environments (58 sites) than into freshwater ones (49 sites). However, in 2016 and 2019 more discharges were into freshwater environments than salt/brackish ones.

Figure 5. Numbers of Sites with Effluents with Toxicity Tests per Type of Receiving Environment

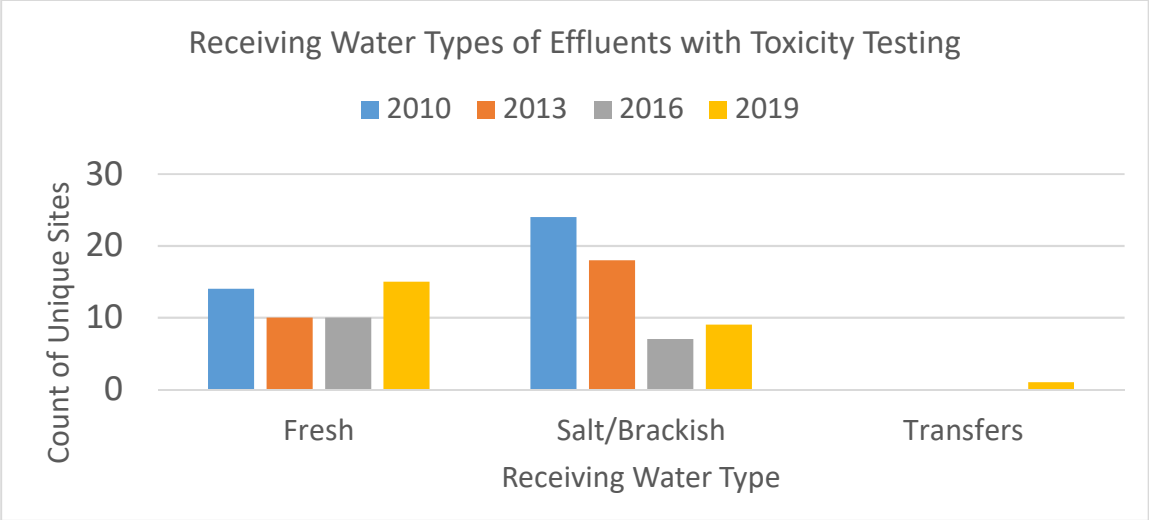
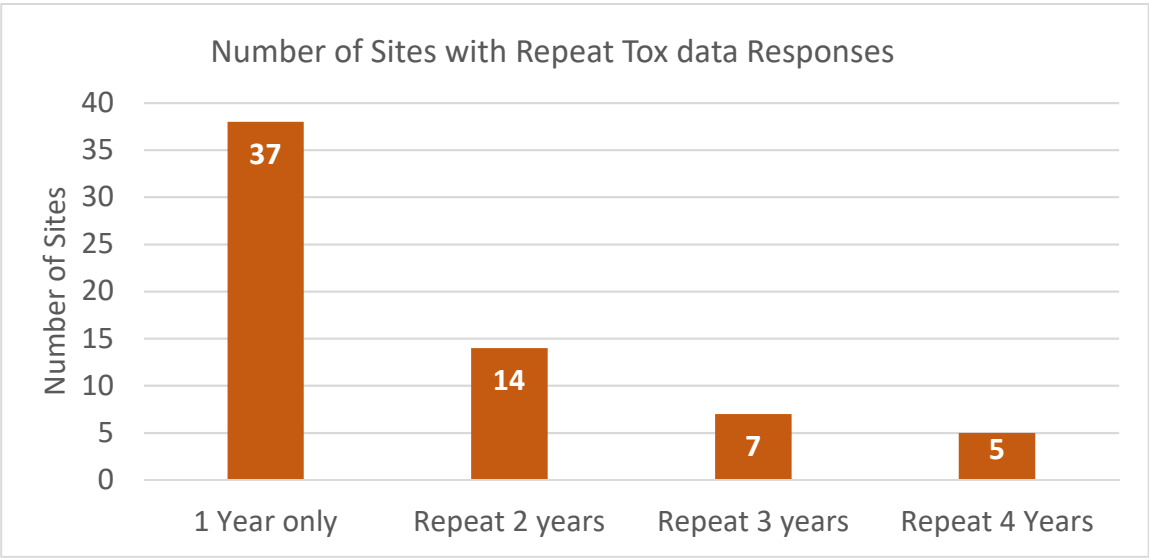


Figure 6 shows the number of sites that reported toxicity data for one or more years out of the four survey years. Underlying data shows an apparent reduction in the number of refineries that reported toxicity data from 2010 to 2019 (37 in 2010 and 24 in 2019). 37 refineries only provided toxicity data in one of the four surveys, and only five provided data for all the surveys.

Figure 6. Number of Sites with Repeat Toxicity Data Responses



In addition, when looking at those sites with only one year of data reported across the four available years (i.e., not closed) we can see that:

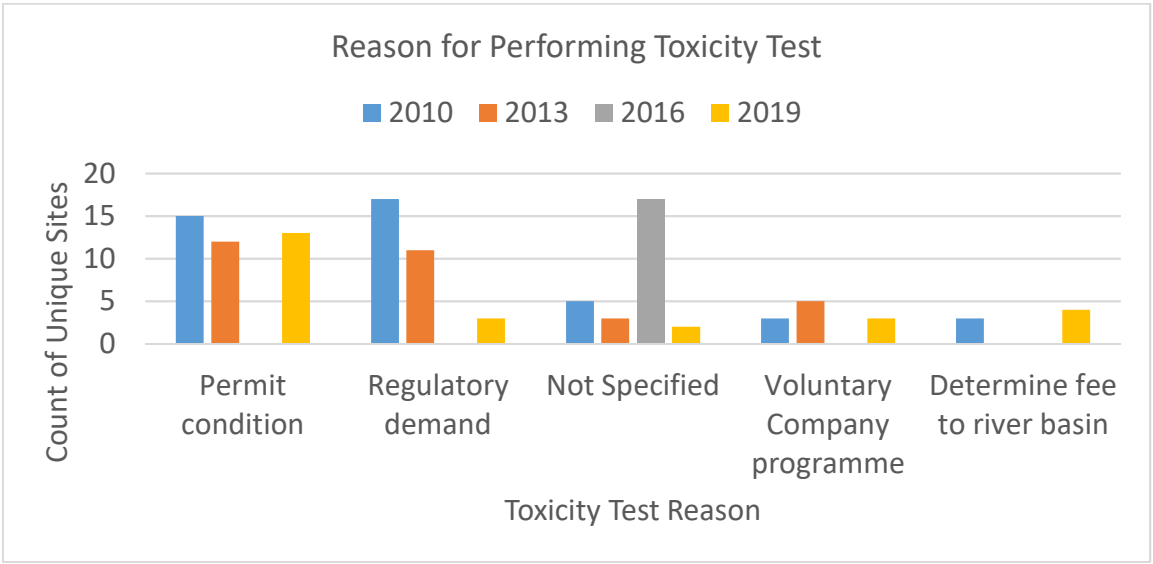
- 20 of them could have submitted data for all 4 years.
- 4 of them could have submitted tox data for 3 years.
- 4 of them could have submitted data for 2 years; and
- 9 could have submitted data only for one year.

Considered together, the above figures seem to suggest the reduction in reported toxicity data may be the result of multiple reasons including: the reduction of the total number of available refineries; a reduction of refineries responding to the survey; and a reduction of those refineries that responded that also provided toxicity data.

2.2. REASON FOR PERFORMING TOXICITY TESTS

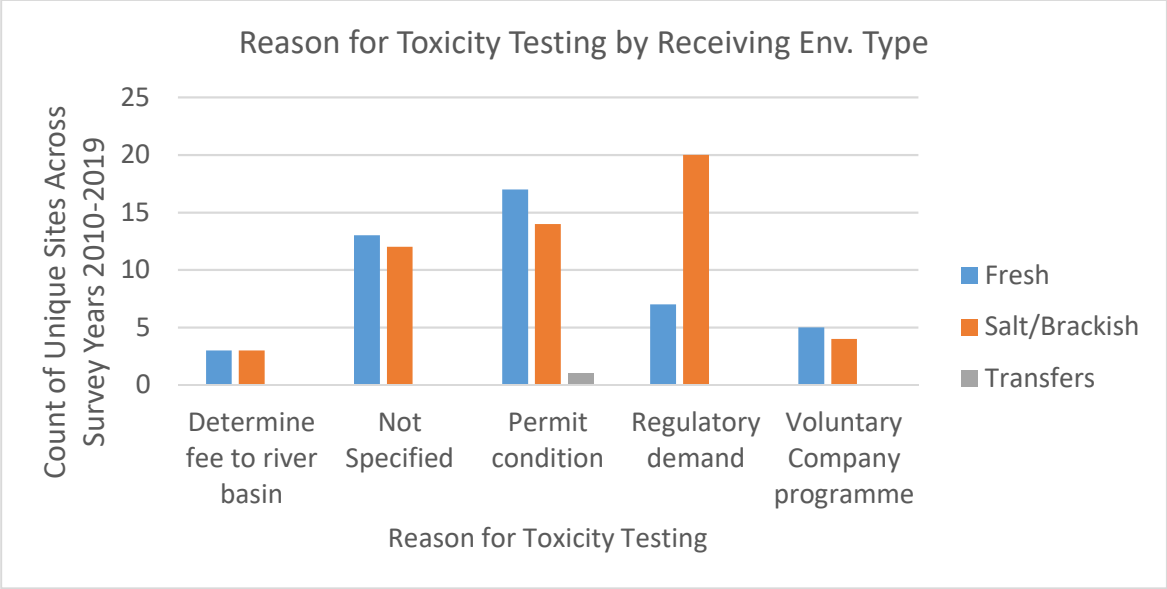
The main reasons given for toxicity testing in 2010 and 2013 were regulatory demand and to satisfy permit conditions. Permit conditions was also the main reason given in 2019 (**Figure 7**). In 2016, the question for the reason for toxicity testing was omitted from the questionnaire. Some sites continued to perform toxicity testing on a voluntary basis from 2010 up to 2019.

Figure 7. Reported Reasons for Performing Toxicity Tests



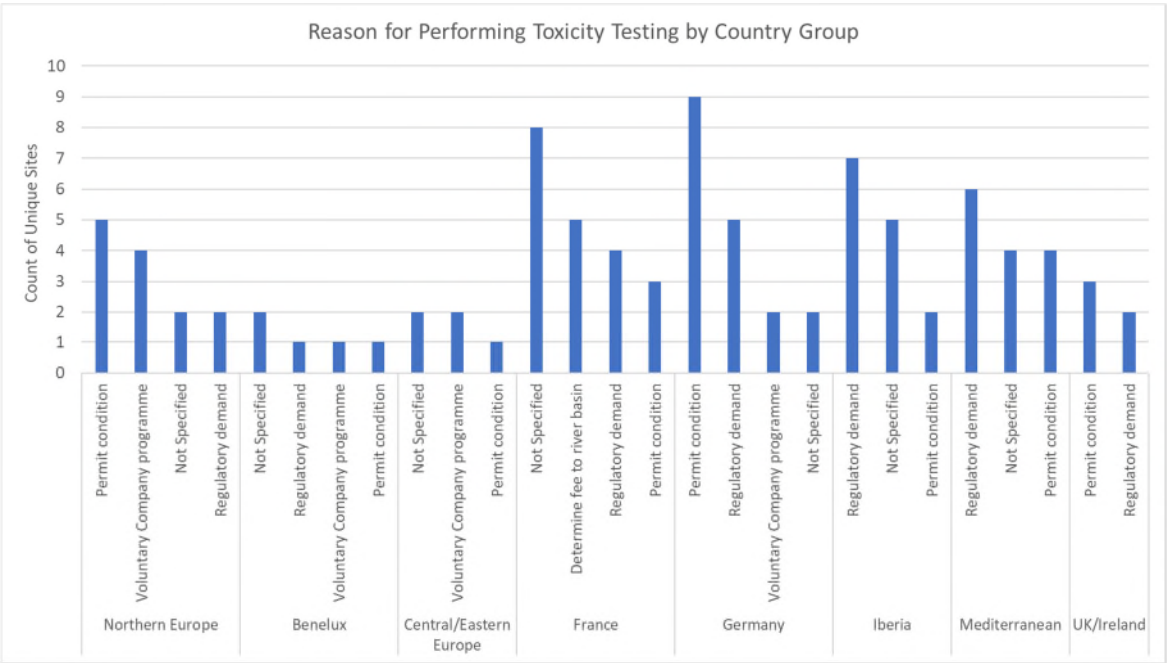
When the receiving environment is considered, the reasons reported seem to apply equally to both freshwater and salt/brackish environments, with only regulatory demand reported mainly in association with a salty/brackish receiving environment (see **Figure 8**).

Figure 8. Reason for Toxicity Testing by Receiving Environment Type



As shown in **Figure 9**, permit conditions and regulatory demand seem to be the main reasons to perform toxicity testing in most country groups. Toxicity testing to determine the discharge fee to a river basin only applied to France, likely due to France's environmental policies. Voluntary company programme testing was performed in Northern Europe, Benelux, Central Europe, and Germany.

Figure 9. Reasons for Performing Toxicity Testing by Country Group



2.3. TYPES OF TESTS PERFORMED

As shown in **Figure 10**, *Daphnia magna* was the most performed test in the four survey years considered in this report. This was followed by luminescent bacteria (*vibrio fischeri*) and fish eggs (*Danio rerio*). Some of the tests were applied to both freshwater and salt/brackish receiving environments, as shown in **Figure 11**. While luminescent bacteria (*vibrio fischeri*) can be applied to both receiving environments, benthic invertebrate is applicable in freshwater environments only. As shown in **Figure 12**, however, this test was also used in a marine environment, in Iberia.

Figure 12 also shows that country groups who are not landlocked such as Northern Europe, France and the Mediterranean mainly performed toxicity testing on effluents discharged into salt/brackish water as their refineries are likely to be coastal.

Figure 10. Number of Sites per Toxicity Tests

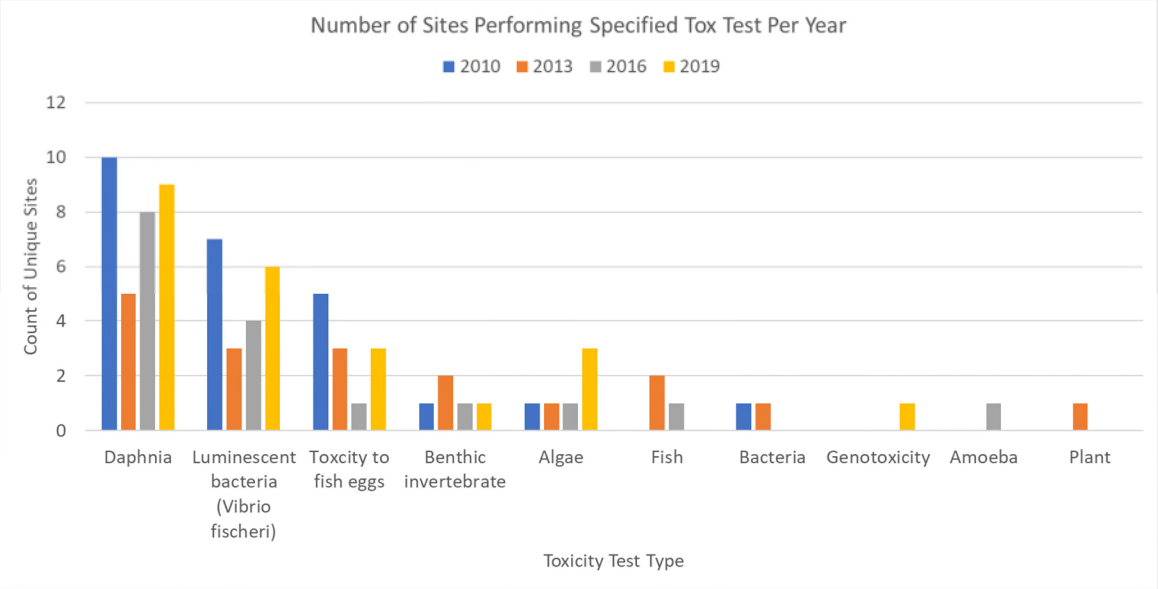


Figure 11. Toxicity Test Types and Receiving Environment Types

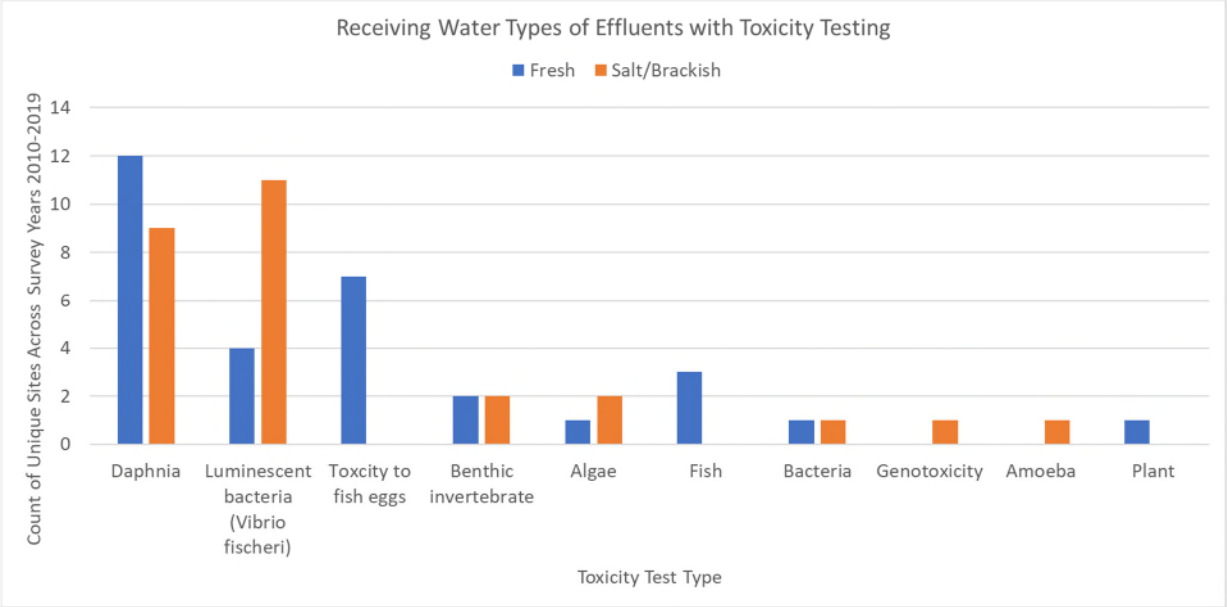
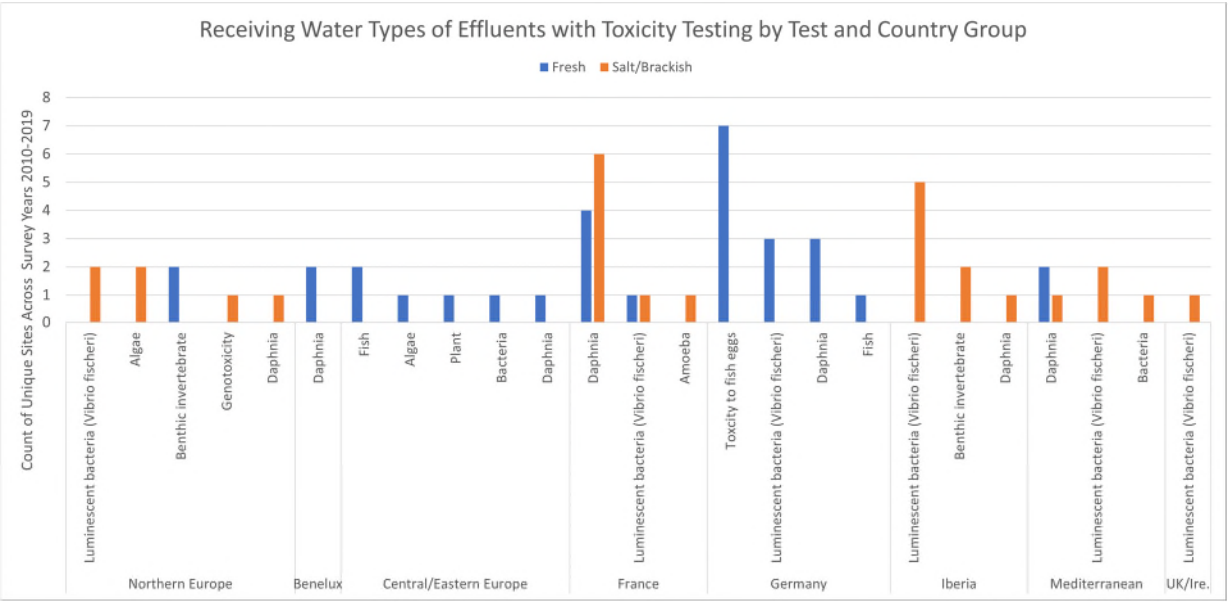


Figure 12. Toxicity Test Types by Receiving Environment Type and Country Groups



Before 2019, the surveys allowed for free-form entry of toxicity test being performed and data could also be extracted from notes providing little information on the standards used when performing toxicity tests. The 2019 survey provided, for the first time, the specific toxicity test standards which could be selected by the reporting refinery. The reported standards are shown in **Table 2**, and they correspond to tests for acute conditions. The inclusion of this field in the survey will further help Concawe to better understand the procedures for toxicity testing that their members are using.

Table 2. Test Types Reported in 2019

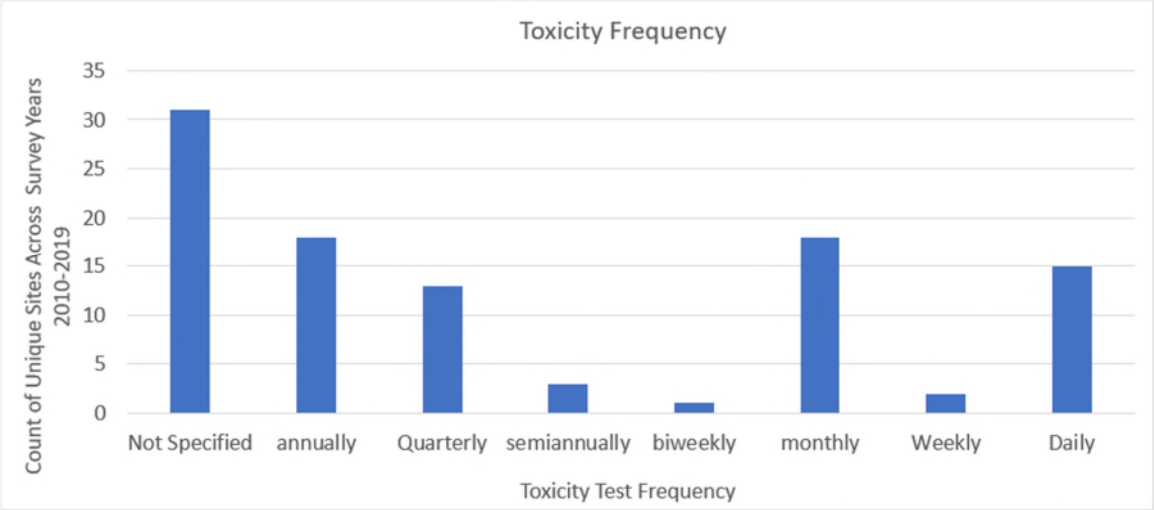
Test types as reported in 2019
Algae - EN ISO 8692
Daphnia (Daphnia magna Straus) - EN ISO 6341
Daphnia (DIN 38412-30)
Fish eggs (Danio rerio) - EN ISO 15088[1]
Fish eggs (DIN 38415-6)[1]
Toxicity for fish eggs (DIN 38 415-T 6)[1]
Luminescent bacteria (Vibrio fischeri) - EN ISO 11348

[1] EN ISO 15088 is the is the most recent protocol for fish egg test. It superseded the DIN 38415-6. The three fish egg test protocols listed in the table are nearly identical.

2.4. FREQUENCIES OF TESTS PERFORMED

Figure 13 shows a combined 2010 to 2019 summary of the toxicity test frequencies undertaken during the four water surveys. As reported previously in the Toxicity review report 2018, the questionnaire in 2010 did not specifically request the responders to indicate the toxicity testing frequency being employed for each effluent stream. The scant information available for 2010 was provided from some respondents in the notes section. From 2013, the survey requested that responders to provide the frequency of toxicity testing. As indicated previously, in 2016 the toxicity section of the survey was not required to be entered and therefore the number of answers that year were lower.

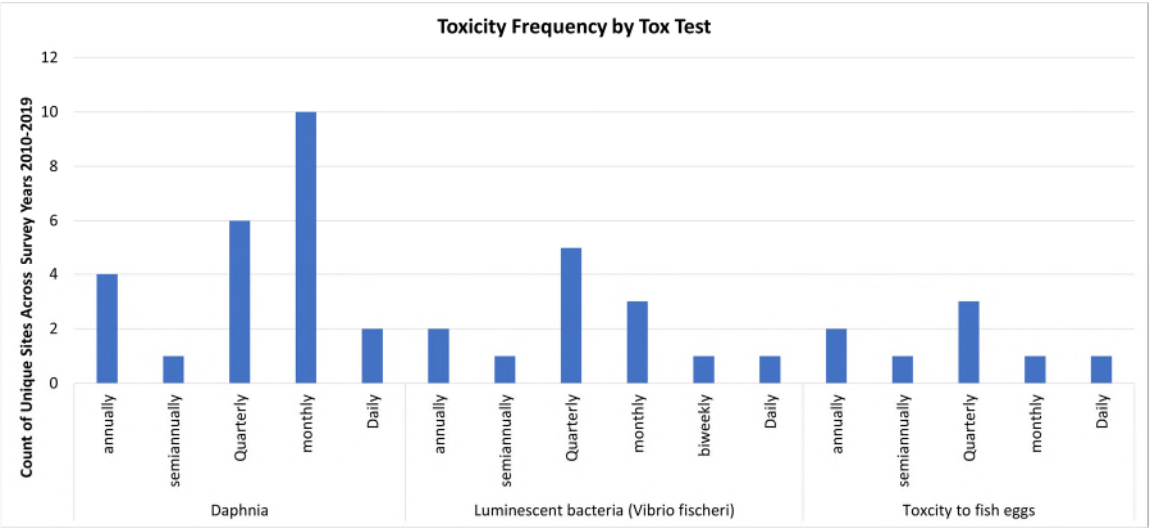
Figure 13. 2010-2019 Toxicity Testing Frequency



When the answers from the four surveys are combined, 62% of the respondents provided a frequency in their answers, while the remaining respondents did not specify a frequency. Annually and monthly testing were the most common monitoring frequencies reported, followed by quarterly.

As shown in **Figure 14**, when segregated by the top three toxicity tests performed, it appears that Daphnia tests were most commonly performed monthly, whereas *vibrio fisheri* and toxicity to fish egg tests were most often performed quarterly.

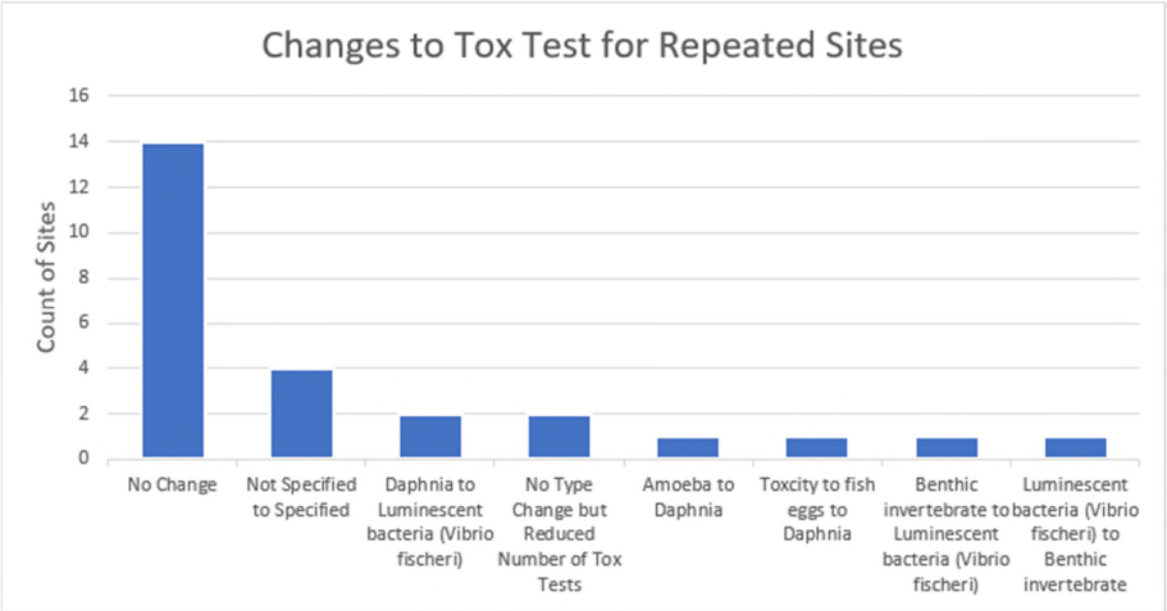
Figure 14. 2010-2019 Toxicity Testing Frequency



2.5. CHANGES TO TOXICITY TESTS

The changes in the toxicity tests across the four survey years is presented in **Figure 15**. The majority of refineries show no change to the toxicity testing performed. Of those that did change, data shows a change from not specified to naming a specific test, a reduction in toxicity test, and some changes in testing methods. The reasons for the changes made are not known.

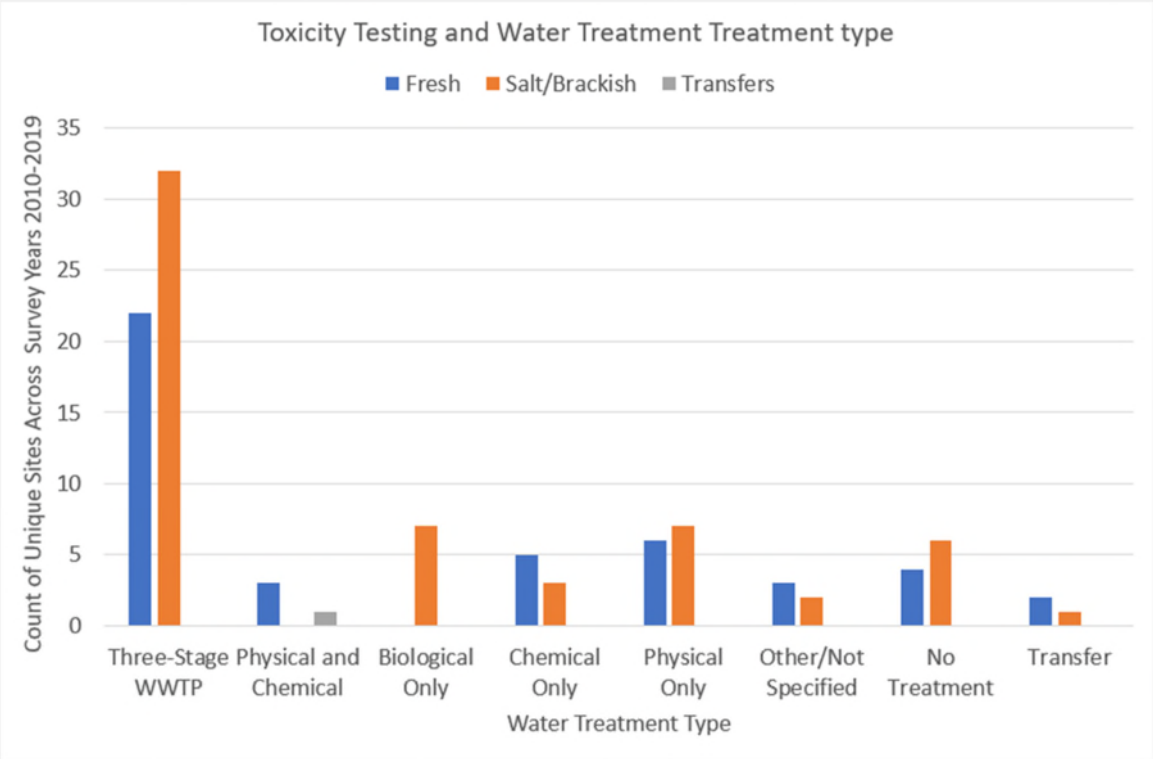
Figure 15. Changes to Toxicity Tests for Sites with Repeated Tests



2.6. TOXICITY TESTING AND EFFLUENT STREAM TREATMENT PROCESS

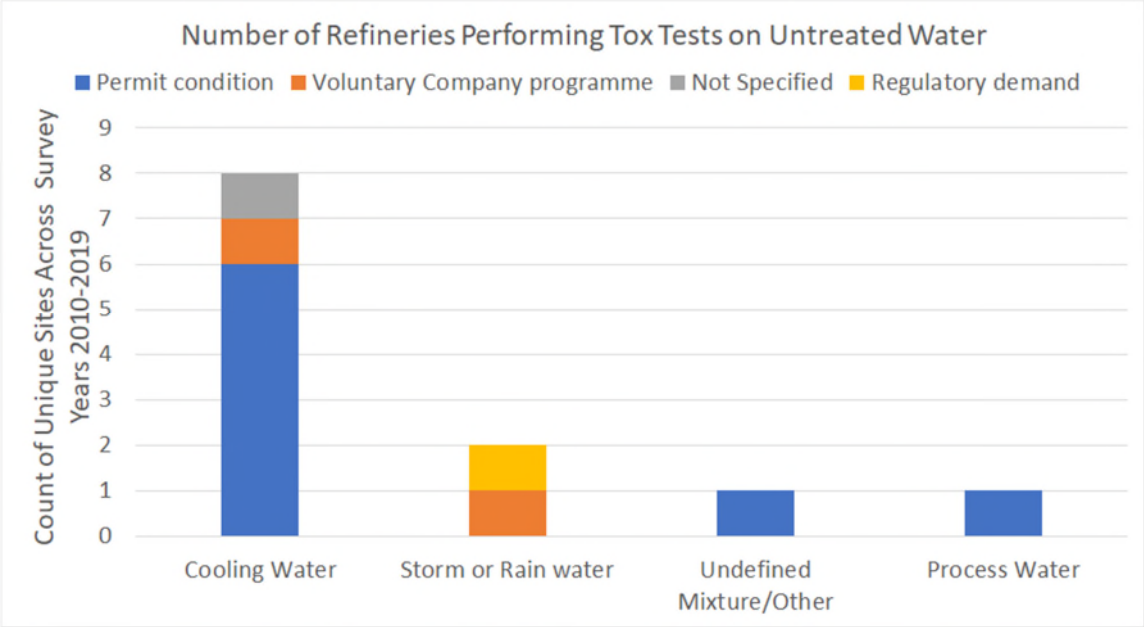
Figure 16 shows the number of refineries with toxicity testing per type of effluent treatment. The four years' data show that effluent waters which were first treated with a 3-stage wastewater treatment process were considerably more often tested for toxicity compared with effluents going through a single treatment step or untreated. This coincides with the data included in the Concawe 2018 Toxicity report (Concawe 2018) and with the fact that 3-stage wastewater treatment is the most common treatment process utilised by the industry in 2010 to 2019 time-frame.

Figure 16. Toxicity Testing and Water Treatment Type



Interestingly, there were several sites which conducted toxicity tests on waters that had no form of treatment. Looking specifically at these sites, it was evident that the majority of the untreated water streams with toxicity testing were related to cooling water followed by storm/rain water, as shown in **Figure 17**. This figure also indicates that the predominate reason for the toxicity testing for the untreated water was to satisfy permitting conditions. Germany had the most toxicity tests on untreated waters in the 2010 to 2019 period.

Figure 17. Number of Refineries Performing Toxicity Tests on Untreated Water



3. CONCLUSION

Data from the 2010, 2013, 2016, and 2019 Concawe refinery effluent surveys has been used to assess the use of toxicity test on effluent streams. In all four survey years at least one refinery within the eight country groups reported using toxicity tests, with the exception of the UK and Ireland for the 2016 or 2019 questionnaire. This indicates that toxicity testing is widespread.

The overall rate of responses to the surveys decreased over the four reporting years (from 89% to 64%) while there was no meaningful change in percentage of participating refineries reporting toxicity data in the same time frame (remaining at approximately 37% in 2010 and in 2019). As the 2016 survey did not require refineries to enter toxicity data, there was a much lower percentage of toxicity data responses that year (24%).

The overall decrease in responses shown above is likely due to a number of factors including refineries closure, less refineries responding to the survey and finally less refineries reporting toxicity data. The surveys also indicate a larger number of outfalls than refineries and that not all outfalls are tested for toxicity.

The survey responses indicated that the primary reason for toxicity testing was to satisfy permit conditions and regulatory demand. However, across multiple country groups toxicity testing was voluntary performed by some refineries. France was the only region where toxicity testing was performed to determine the discharge fee.

Daphnia magna was the predominant type of toxicity testing across all four survey years followed by luminescent bacteria *(Vibrio fischeri)*. Both tests were performed on fresh water and salt/brackish receiving environments. Though toxicity to fish eggs was also a common type of toxicity test, this was only performed on fresh water receiving environments. Annually and monthly toxicity testing frequency were the most commonly reported across the 2010 to 2019 surveys.

Most refineries reported no change over the years as to the type of toxicity test performed, followed by a change from not specified to naming a specific test. The rest reported a change from one type of test to another, but to no particular type over time.

Improvements to the questionnaire such as the use of drop-down fields and the inclusion of the specific toxicity tests standards has helped to standardize further the survey and to gather further data for analysis.

4. GLOSSARY

DIN	German National Standard
EN	European Standard
EU	European Union
EU-28	Abbreviation of European Union (EU) which consists of a group of 28 countries
ISO	International Organisation for Standardisation

5. REFERENCES

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2. Concawe (2020). 2016 Survey of Effluent Quality and Water Use at European Refineries

3. Concawe 2019 survey data

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