

EUROPOP

FINAL REPORT



CLEEN
Chemical Legislation European
Enforcement Network

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- Sabine Cladrowa, Austria
- Simone Fankhauser, Austria
- Tina Klarskov, Denmark

Contact: CLEEN Secretariat and EUROPOP Working Group

www.cleen-europe.eu

Heribert Bürgy: Heribert.Buergy@bag.admin.ch

Viktoras Šeškauskas: <mailto:zucht.gerhard@baua.bund.dev.seskauskas@aaa.am.lt>

Sabine Cladrowa: Sabine.Cladrowa@umweltbundesamt.at

Simone Fankhauser: Simone.Fankhauser@umweltbundesamt.at

Tina Klarskov: tikla@mst.dk

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I. INTRODUCTION and SUMMARY

I.1 About CLEEN

CLEEN is a voluntary network of chemical inspectorates in European Union, Norway and Switzerland that aims to coordinate the enforcement of EU chemical legislation by developing common strategies and tools for the inspectors in the member countries. It is basically a forum for information exchange and it performs enforcement projects as proposed by its members. As enforcement is under the responsibility of the member states, the cooperation of the national chemical inspectorates in the European area is absolutely necessary in view of the rules of the single market and the EU-wide economy. The aim is to consolidate and intensify such co-operation so that compliance with chemical legislation can be improved for the protection of human health and the environment.

I.2 About EUROPOP

HCB was widely used from the 1940s to the late 1970s as a fungicide on grain seeds such as wheat. In the USA the last registered use as a pesticide was voluntarily canceled in 1984. In the USSR HCB was used till 1990 as Hexathiuram and Hammahexane in agriculture, in forestry and for municipal purposes; in the period of 1990–1996 only in Russia remains of these products were allowed for application. HCB was also used to make fireworks, ammunition, and synthetic rubber. The aim of the project was to foster a great degree of compliance according to the present EU legislation and possibility of risk reduction to man and the environment by taking administrative measures to withdraw products containing HCB from the European market.

HCB is classified as Carc. 1B, H350; STOT RE 1, H372; Aquatic Acute 1, Aquatic Chronic 1, H400, H410 according to the CLP Regulation and Carc. Cat 2; R45; T; R48/25; N; R50-53 according to the Dangerous Substances Directive.

Before setting up the EUROPOP project, control activities in fireworks have been undertaken in Denmark in 2008 - 2010 and in Austria in 2009 - 2010. In total 82 samples have been taken, which have all been imported from China. Analyses showed that 25% of the products contained HCB. The contents ranged from 0,005% to 4,4% which is above the quantification limit of 0,0005% and above the level of a trace contaminant. After the undertaken national control activities, Austria proposed a new European enforcement project to the CLEEN members during the 11th CLEEN conference in September 2010, the EUROPOP project.

Within the CLEEN network, originally nine countries agreed to participate in the EUROPOP project: Austria, Belgium, Denmark, Estonia, Germany, Iceland, The Netherlands, Sweden and Switzerland, later on also Finland and UK provided results of their enforcement activities. I.e. that in the end 11 countries submitted the results of their HCB enforcement activities. The project working group consisted of Austria and Denmark and the project management was carried out by Austria.

In total, 410 samples were analysed from which 41 samples showed violations, i.e. they contained HCB in concentrations above 50 mg/kg, which was identified as the Limit value within the EUROPOP project. This value is based on the waste provisions following Article 7 of the POPs regulation: Article 7 states that all efforts have to be undertaken to avoid contamination of waste above the limit value of 50mg/kg. 81 samples were below the Limit value, but above the Quantification limit of 5 mg/kg HCB. 288 samples showed a HCB content below the Quantification limit.

II. BACKGROUND

II.1 Background and legislation

The objective of Reg. (EC) No 850/2004, the so called “POP Regulation” is to protect human health and the environment from persistent organic pollutants by prohibiting, phasing out or restricting the production, placing on the market and use of substances. Under the scope of the POP regulation are substances which are subject to the

- Stockholm Convention on Persistent Organic Pollutants or the
- Protocol on Long-Range Transboundary Air Pollution on Persistent Organic Pollutants

In Article 3 it says that “The production, placing on the market and use of substances listed in Annex I, whether on their own, in preparations or as constituents of articles, shall be prohibited”. Annex I of the regulation contains the list of substances subject to prohibitions. One of these substances is Hexachlorobenzene (HCB).

Exempted from this prohibition are those substances listed in Annex I which are substances occurring as an unintentional trace contaminant in substances, preparations or articles.

As a result of the investigations of Austria and Denmark it has been shown that HCB present in fireworks has a material impact on HCB-concentrations in ambient air.

Today the most common method of supplying chlorine or hydrogen chloride in colored pyrotechnic flames is through the use of a chlorine donor, sometimes also called a color enhancer. This is a chlorine rich chemical compound such as hexachlorobenzene (HCB) or polyvinyl chloride (PVC) that reacts in the flame to release chlorine or hydrogen chloride, such as suggested in equations 1 and 2 respectively.



Table 1 List of the most common chlorine donors, along with their percent chlorine by weight

Chlorine Donor	Formula	Chlorine (wt. %)
Dechlorane	$\text{C}_{10}\text{Cl}_{12}$	78
HCB	C_6Cl_6	74
Saran®resin	$(\text{C}_2\text{H}_2\text{Cl}_2)_n$	73
BHC (benzene hexachloride)	$\text{C}_6\text{H}_6\text{Cl}_6$	73
Parlon®	$(\text{C}_5\text{H}_6\text{Cl}_4)_n$	68
Chlorowax®	Variable	40-70
PVC	$(\text{C}_2\text{H}_3\text{Cl})_n$	57

III. PROJECT DESCRIPTION

III.1 Project set-up

As the EUROPOP project comprises inspection as well as sample analysis, the preparation of inspectors and laboratories are carried out in three phases, a preparation phase, an inspection phase and a reporting phase. The project has been presented at the 11th CLEEN conference in September 2010 in Sucevita, Romania. All Member States were invited to participate in the project. It was agreed that the project will be managed by a working group consisting of Austria and Denmark. Austria took the lead and – supported by Denmark - acted as focal point for all administrative tasks including coordination for elaboration of the inspection manual, drafting the time schedule and handing over the manual, as well as distribution of other joint/general information to the participating countries.

1. Preparation phase

- Agreement of the project at 11th CLEEN conference
- Clarification about participation in written procedure until end of November 2010
- Locate/inform involved authorities (laboratories)
- Preparing for inspection, including training for inspectors
- Preparing laboratories for testing and taking samples

2. Operation phase

- Inspection and taking samples
- Sending samples to laboratories
- Chemical analysis of taken samples: based on waste management provisions (Annex IV POP regulation) HCB concentrations of 50 mg/kg (= 0,0005 % w/w) and more are considered as violations of the regulation, concentrations below 50 mg/kg are considered as trace contaminations

Product types

- Fireworks

Companies

- Importers (e.g. from China)
- Manufacturers (within the EU)

3. Reporting phase

The final report was prepared by the WG in summer 2012 after all results concerning the inspection of the companies, analysis and problems have been submitted by the Member States. The aim was to submit the results in the checklist format (see Annex to the project manual as well as to the final report), filled in by the inspectors and sent to the working group, which was done by most participants. The results together with the final report were presented at the CLEEN conference in autumn 2012. An overview of the inspections and the results is given in Annex I to this report, some general remarks can be found in section III.3 Results.

III.2 Analyses of samples

The laboratories have not yet dared to crush the stars (HCB is presumed to be in the effects only) before extraction, so they take typically all the effects in one pyrotechnic unit in a 100 ml Bluecap add 50 ml water to dissolve/soften any starch based “glue” and let it stand with a magnet mixing for a period of 45 – 60 min. If there is much in the liquid phase they centrifuge it and discard the water. Then they extract same way with acetone (again about 50 ml) in the bluecap bottle, and carefully crush by means of a glass spatula any balls left and then they centrifuge and decant the acetone into 100 ml flasks that are filled to the mark. They tend to centrifuge because filters easily stop.

If they dare crush the stars they could avoid the water step, however, some stars could not be crushed in acetone alone and then they were faced with the question whether they all did get out. With the stars “dissolved” they are quite certain that they have the major part of any HCB present. This acetone is then analysed by GCMS, where HCB is easy to identify (Denmark: ions 284+264+288; Austria: ions: 284+282+286+249+251), and it has no tailing. They use an ordinary column RTX 5MS 30 m*0,25 mm (in Denmark), column DB5 MS, 60m, 0,25 µm Film, 0,25 mm ID (in Austria) with a rather fast temperature program, split 100:1. They can certainly see levels of 10 ppb HCB in the acetone (10 ug/ml).

Checklists (see Annex II) have been compiled in order for the inspectors to collect information on company data, product data, data on the analysis such as laboratory name and results (Quantification limit (QL) of 5 mg/kg and Limit value (LV) of 50 mg/kg) as well as on any consequences in case of violations.

III.3 The results – a first overview

A detailed overview of the reported results can be found in Annex III of this report. Some general remarks partly including summaries of the activities are presented in this section.

Austria

In Austria the regional Chemical Inspectorates within the federal states are responsible for enforcement of the chemical legislation. The samples collected by the inspectors were sent for testing to the laboratory of the Environment Agency Austria (Umweltbundesamt) in Vienna. Analyses of 22 samples taken in 2011 have been performed, of which 18 were below the QL (Quantification limit) of 5 mg/kg, 4 samples were below the LV (Limit value) of 50 mg/kg, but above the QL and no sample showed a HCB content above 50 mg/kg. It was not reported whether the companies acted as manufacturer or as importer.

Belgium

Belgium tested 11 samples and reported the results via the checklist. 9 samples were below the QL of 5 mg/kg while 2 samples were above the LV of 50 mg/kg. All inspected companies imported their products; none of the companies manufactured the fireworks themselves. In the cases where the samples were above the LV, legal action has been taken according to national legislation which is a warning according to the national law on product standards (sales stop + recall). Next autumn it is planned to perform a new inspection and if HCB is again detected in amounts over the LV an official report will be submitted to the public prosecutor.

Denmark

In Denmark 29 products of consumer fireworks have been checked. All in all 74 samples corresponding to 29 products were tested. The results show that 41 samples were below the QL of 5 mg/kg, 24 samples were below the LV of 50 mg/kg but above the QL and 9 samples were above the LV.

All 29 products were imported from China. 5 products had a HCB content above the LV of 50 mg/kg. In 4 cases the importer has been banned from selling the product and required to dispose of the product in a legal manner according to Danish legislation. In one case no legal action has been taken because the result showed a HCB content of 62 mg/kg, which is slightly above the limit value of 50 mg/kg.

Estonia

In Estonia 10 samples have been checked which were taken from imported products only. None of the 10 cases showed a HCB content above the QL of 5 mg/kg.

Finland

Finland reported of 18 samples that have been tested for the HCB content and which were taken from imported products only. 14 samples were below the QL of 5 mg/kg while 2 samples were below the LV of 50 mg/kg but above the QL and 2 samples were above the LV. Furthermore, Finland provided the following report:

Introduction

In 2009 Danish authorities reported via RAPEX system that according to the tests carried out in Denmark several fireworks on the Danish market contained quite large amounts of HCB. HCB was detected in effect charges intended to produce colourful light effects. None of the fireworks in concern appeared to be on the market in Finland according to the register of approved fireworks. However, Finnish environment institute (SYKE) that was in charge of enforcing the POP Regulation in Finland sent a letter to all importers of fireworks reminding of the ban to use HCB and urging the importers to take actions to make sure that their products conform to the provision.

However, just before the end of 2010 rose doubts based on qualitative test results on two products that there might be fireworks on the Finnish market in which HCB had

possibly been used. To get realistic picture of the situation in Finland it was decided to carry out a testing project where representative sample of fireworks potentially containing HCB were collected and analysed to find out if they contained the substance. Altogether 18 products from all four importers operating in Finland were tested. All the products were manufactured in China.

Sampling and testing

Samples were collected during spring 2011 by the Finnish Safety and Chemicals Agency (Tukes) as part of Agency's general fireworks surveillance visits to the storage premises of the Importers. Analyses were carried out by the laboratory of the Finnish Technical Research Centre of the Defence Forces. The laboratory had to develop a proper quantitative analysis method before they were able to carry out the task. Consequently the samples were analysed in late autumn in 2011. The test method used was based on gas chromatograph combined with mass spectrometer. The lowest concentration of the reference sample was 0,05 ppm. Test samples were taken from different parts of the fireworks and the total number of individual analyses made was 127.

Conclusions

There was only one product where not even traces of HCB were detected. In 13 fireworks HCB was detected in at least some part of the product but concentrations were so low that they were interpreted as unintentional contamination (article 4 in POP Regulation) and no further actions were taken except that the importers were informed of the results and urged to remind the manufacturers of the HCB ban. Two products contained so much HCB (181 ppm and 274 ppm), that further actions were taken by Finnish environment institute. In all cases concentrations of HCB were deemed to be below the limit in the restriction no 28 in Annex XVII in REACH Regulation.

During the dialog with the importers it turned out that the consignments of both products imported for the use in the New Year's Eve 2011 did not contain HCB any more in excessive amounts. For one of the product it could even be verified by further testing by Tukes. That being the case no withdrawals from the market and other legal actions were taken by SYKE.

Germany

Germany tested 182 samples corresponding to 98 products. 126 samples were below the QL of 5mg/kg, 38 samples were between the QL and the LV of 50 mg/kg and 18 samples were above the LV. As far as the products are concerned, 15 rockets and 2 batteries were above the LV of 50 mg/kg, while 20 rockets and 9 batteries were between the QL and the LV.

Iceland

All in all 22 samples corresponding to 9 products were tested by Iceland of which 17 were below the QL of 5 mg/kg, 1 sample was between the QL and the LV of 50 mg/kg and 4 samples showed a content of HCB above the LV. The following report provided by Iceland gives a comprehensive overview of the activities taken:

Nine samples of fireworks, batteries and rockets, were analysed for the content of HCB. One sample was collected from each of the importers in 2011. They were collected from 16th to 28th December. The reaction of the importers was very positive in general. They were all cooperative and aware of the probability of a possible HCB content in fireworks. All of the fireworks imported last year come from China. Two samples did not comply with the POPs regulation and contained HCB in the colour effects (600 mg/kg (one out of two effects above the limit of 50 mg/kg) and 480 mg/kg (all three effects above the limit)). Three samples contained less than 2 mg/kg and four did not contain any (detection limit of 0,25 mg/kg). All samples with HCB content less than 50 mg/kg are considered to be in accordance with regulation (EC) 850/2008. Legal action is not possible because of the limits are uncertain but all remaining items of the illegal fireworks are considered to be hazardous waste and shall be disposed according to article 7 of regulation (EC) no. 850/2004. Some of the importers had an excuse in advance from the manufacturers like: "HCB is all around here in the air, water and soil", "HCB traces are on the manufacturing site because it is still used in fireworks for markets in Asia". Higher concentration of HCB has been measured in Icelandic biota than can be explained by long range transport from distant sources. HCB was also measured in ambient air in the new year of 2011 in 900 time's higher concentrations than background levels. Fireworks play an important role in the New Year's celebration in Iceland. They may only be fired by the public

between December 28th and January 6th. Four of the importers are non-profit organisations whereof one has by far the largest market share. The rest are companies which are only active temporarily for a few days around the turn of the year. Imports of fireworks: 420 tons in 2009, 500 tons in 2010, 550 tons in 2011 – increasing since 2008 after a significant decrease from the previous year.

The Netherlands

The following report on HCB analysis in ‘consumer’-fireworks (scope 2010 and 2011) was provided by the Netherlands:

2010

In 2010 we started with HCB-analysis in ‘consumer’-fireworks in the Netherlands on request of Department ILT (former Department of VROM). At the time the NFI did not have a validated analysis for HCB’s in pyrotechnic mixtures. So the Department of ILT and NFI agreed to perform only a qualitative analysis on HCB’s. Within the contract a maximum of 10 fireworks articles would be selected during the physical testing of these articles based on colour-performance. At first 8 fireworks articles which spread blue-coloured effects where selected.

The results of these analyses would give a global indication on the use of HCB’s in fireworks articles which were imported in the Netherlands.

For the analysis on HCB’s in pyrotechnic mixtures, a GC-MS method was used.

The results of 2010:

Of the 8 selected fireworks-articles only one fireworks article proofed to be positive on the analysis for HCB.

The article proofed to be positive on HCB, is not a ‘CE’ –article, but a battery of single shot tubes, 16 shots, namely:

Item name: Red Shadow, Masterline

Item no.: RVW304

Imp. Rubro

Prod.year: 2009-10

2011

Based on the results of 2010, the implementation of the CE-regulations, some foreign RAPEX-notifications, the Department of ILT asked the NFI to set up a validated quantitative analysis on HCB in pyrotechnic mixtures.

To be able to set up a validated analysis, approximately 11 fireworks articles were selected for this project. The NFI is now able to perform a quantitative analysis on HCB in pyrotechnic mixtures. The method has been validated.

As a result the NFI reports when within three levels on HCB concentration in pyrotechnic mixtures:

- HCB concentration is below detection limit*
- HCB concentration is between detection limit and the allowed concentration (contamination level) (50mg/kg)*
- HCB concentration is above the allowed concentration (contamination level)*

During the physical test in 2011 also three articles were selected for a quantitative analysis on HCB. The results are given below.

Black Star assortment, article no. 5540, imp. GBV, prod. Year 2011, CE-selection-pack.

From this selection-pack, the article Black Star 1, a fountain has been analyzed. In the pyrotechnic mixture of this fountain, no HCB has been demonstrated (below detection-limit)

Magnum Night Display, article no. VC 92, imp. Broekhoff Fireworks International, prod. Year 2011, CE-selection-pack.

From this selection-pack, the article Night Display, art.no. 1911-7, CE-336-F2-27331-1911-07, a battery of single shot tubes, of 9 shots, has been analyzed. In the pyrotechnic mixture of this battery, no HCB has been demonstrated (below detection-limit)

High Five, Article no. VC 111, imp. Broekhoff Fireworks International, prod. Year 2010, CE-selection-pack.

From this selection-pack, the article Star Tracer, art/no. VC111-02, CE-336-F2-27335-855B, a battery of single shot tubes, of 16 shots, has been analyzed. In the pyrotechnic mixture of this battery, a concentration of HCB has been demonstrated between the detection limit and the allowed limit of 50mg/kg (below contamination-level)

Sweden

40 samples corresponding to 12 products have been analysed in Sweden of which 28 samples were below the QL of 5 mg/kg. 2 samples were below the LV of 50 mg/kg but above the QL of 5 mg/kg whereas 10 samples were above the LV. The samples have been taken from companies acting as importers. As regards the 3 products which showed a HCB content above the LV a report to the Swedish prosecution agency has been sent.

Switzerland

In Switzerland 47 firework articles have been collected for the determination of HCB. The articles included roman candles (17 %), rockets (20 %), fountains (23 %), batteries (23 %) and bombs for use by professionals (17 %). The origin of the products was China (72 %), Germany (19 %) and Switzerland (4 %). After careful delaboration of the firework articles by specialists, the effect compositions have been analyzed for HCB. If the firework article contained several effect compositions they were pooled to one composite sample for analysis. Overall 51 samples have been checked of which 46 samples were below the QL of 5 mg/kg. 5 samples showed a content of HCB over the QL but still below the LV of 50 mg/kg. No cases of HCB above the LV could have been detected. As none of the samples showed a HCB content above the LV, no further action has been taken.

In addition to the market survey an air pollution measurement campaign was conducted on August 1, 2011, the Swiss national holiday, when firework is used extensively. Ambient air was collected by high-volume sampling at the NABEL station "Kaserne" in the city center of Zurich on July 24, August 1 and 2, and August 14. The air samples were analyzed for chlorobenzenes (tetra-, penta- and hexachlorobenzenes), chlorophenols (tri-, tetra-, and pentachlorophenols) and dioxins (polychlorinated dibenzo-*p*-dioxins and dibenzofurans, PCDD/F). The concentration of HCB in the air was around 60 pg/m³ on the days when no firework has been used. This concentration corresponds to background levels measured in Central Europe. Air concentrations of the other chlorobenzenes and chlorophenols were between 10 and 30 pg/m³, air concentrations of dioxins were in the range of 10 to 20 fg I-TEQ/m³. In the night between August 1 and 2, when fireworks have been extensively used, air concentrations up to 350 pg/m³ for the sum of chlorobenzenes, 430 pg/m³ for the sum of chlorophenols and 60 fg I-TEQ/m³ for dioxins have been

measured (3h sampling intervals). For HCB, the highest concentration was 300 pg/m³. In summary it can be concluded that urban air concentrations of HCB and other chlorobenzenes, chlorophenols and dioxins can be elevated by a factor of five during periods of extensive use of fireworks, compared to background concentrations in urban air.

United Kingdom

In the UK 6 samples have been taken and none of those showed a concentration of HCB above the QL of 5 mg/kg, all of the fireworks tested were below 0.15mg/kg HCB. They were imported to the UK from China and are restricted for sale to professionals/public displays only.

IV. SUMMARY and CONCLUSIONS

IV.1 Summary of the results

In total 439 samples have been tested with respect to their HCB content. This number doesn't correspond to the number of products tested, as for some products analyses for several effects were performed. 317 samples showed a HCB content below the Quantification limit of 5 mg/kg, while 77 samples showed a content between the Quantification limit and the Limit value of 50 mg/kg. 45 analyses of the tested samples showed a HCB content of more than 50 mg/kg, i.e. above the Limit value. Table 2 gives an overview of the number of analysed samples, test results and information about legal actions where available.

Table 2 Results of the EUROPOP project analysis on HCB in fireworks

	Samples analysed	below QL	below LV, above QL	above LV	No legal action taken (no of cases)	Legal action taken (no of cases)
		5 mg/kg	5 mg/kg < x < 50 mg/kg	50 mg /kg		
Austria	22	18	4	-	22	-
Belgium	11	9	-	2	9	2
Denmark	74	41	24	9	25	4
Estonia	10	10	-	-	10	-

Finland	18	14	2	2	16	2
Germany	182	126	38	18	?	?
Iceland	22	17	1	4	7	2
The Netherlands	3	2	1	-	-	-
Sweden	40	28	2	10	9	3
Switzerland	51	46	5	-	51	-
United Kingdom	6	6	-	-	6	-
Summe	439	317	77	45	122	13

IV.2 Conclusions

From the 439 samples that were analysed for the HCB content during the EUROPOP project 317 samples showed a HCB content below the Quantification limit of 5 mg/kg, while 77 samples showed a content between the Quantification limit and the Limit value of 50 mg/kg. Only 45 of the tested samples showed a HCB content of more than 50 mg/kg, i.e. above the Limit value. This means that 10% of the tested samples contained HCB above the Limit value. As far as concrete data on the inspections were provided the violations are in range from a slight violation of 62 mg/kg up to quite severe violations of 27.000 mg/kg. The samples analysed during the EUROPOP project show therefore a better compliance of companies than the inspections that have been performed in Denmark and Austria during 2008, 2009 and 2010 which showed a violation rate of 25 % of tested samples. No concrete statement can be made about the proportion of manufactures to importers as not all participating countries provided information on this issue. However, from the information available the number of importers clearly exceeds the number of manufactures.

Annex I: List of participating countries and contact persons

Country	Name	E-mail	Phone/Fax	Department/address
AT	Sabine Cladrowa	Sabine.cladrowa@umweltbundesamt.at	P: +43-1-31304 5651 F: +43-1-31304 5660	Umweltbundesamt Department for Chemicals Spittelauer Laende 5 1190 Vienna, Austria
BE	Michel Leynen	michel.leynen@health.fgov.be Paul.cuypers@lne.vlaanderen.be	P: +32-2-5249564 F: +32-2-5249636	Federal Public Service Health, Food Chain Safety and Environment Place Victor Horta 40 Box 10 B-1060 Brussels, Belgium
DK	Tina Klarskov	tikla@mst.dk	P:+45-72-544524	Danish Environmental Protection Agency, Chemical Inspection Service, Strandgade 29, 1401 Copenhagen, Denmark
EE	Natali Promet	Natali.Promet@terviseamet.ee	P: +37-2-6943533	Health Protection Inspectorate, Estonia
FI	Sari Tuhkunen	Sari.Tuhkunen@tukes.fi	P: +358 10 6052 050	Finnish Safety and Chemicals Agency (Tukes)
DE	Eckard Klein	Eckard.Klein@melur.landsh.de	P: +49 (0)431-988-7104 F: +49 (0)431-988-7239	Ministerium für Energiewende, Landwirtschaft, Umwelt und ländliche Räume Referat V 64 Grundsatzfragen, Stoffbezogener Immissionsschutz, Chemikalien
IS	Haukur R. Magnússon	haukurm@Umhverfisstofnun.is	P: +354-5912000 F: +354-5912010	Environment Agency of Iceland Sudurlandsbraut 24, 108 Reykjavik, Iceland
NL	Arno van Dop	arno.vandop@minvrom.nl	P: 070-456 2176 M: 06-1132 7516 F: 070-456 2651	Organisatie VROM.VI.DU.ZW.VT Postbus 16191 2500 BD Den Haag
SE	Karin Rumar Frida Ramström	Karin.Rumar@kemi.se frida.ramstrom@kemi.se	P: + 46-8-51941252 F: + 46-8-7357698	Swedish Chemicals Agency Box 2 S-172 13 Sundbyberg Sweden
CH	Urs Vonarx	urs.vonarx@bafu.admin.ch		
UK	Christopher Summers	chris.summers@environment-agency.gov.uk	P: +44-3708 506 506 F: +44-0114 289 8353	The Chemical Compliance Team Waste & Industry Regulatory Service Environment Agency

Annex II: Checklist format

1. General information	
Country	
Date of inspection	
2. Company data	
Name	
Company	<input type="radio"/> importer <input type="radio"/> manufacturer
Clients	<input type="radio"/> retailer <input type="radio"/> consumer
3. Product	
Name (trade name)	
Country of origin	
4. Analysis	
Laboratory (name of lab, ref. to the accreditation, ...)	
Result	<u>Concentration of hexachlorobenzene:</u> <input type="radio"/> below QL <input type="radio"/> below LV, above QL: _____ mg/kg <input type="radio"/> above LV: _____ mg/kg Limit value (LV): 50 mg/kg Quantification limit (QL): 5 mg/kg
5. Consequences in case of violation	
<input type="radio"/> No legal action taken. <input type="radio"/> Legal action taken according to national legislation:	