The guar gum case:
Contamination with PCP and dioxins and analytical problems

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Introduction

The Rapid Alert System for Food and Feed (RASSF) of the European Commission received on 24 July 2007 in the evening a notification from the competent authorities of Switzerland concerning a finding of a serious contamination by dioxins and pentachlorophenol in guar gum originating from India. This contamination incident was reported through the RASSF on 25 July 2007 to all Member States by alert notification 2007.0499 (and additions).

Guar gum is an edible thickening agent extracted from the guar bean. India produces approximately 80 % of the world's total production of guar beans. There is a food grade guar gum powder which is authorized as food additive and used as a thickening, emulsifying, binding and gelling additive in a very wide range of foodstuffs. There exists also an industrial grade of guar gum powder for non-food uses.

The contamination levels of dioxins and pentachlorophenol found in July 2007 in certain batches of guar gum were very high. The initially found levels of up to 480 pg WHO-PCDD/F-TEQ/g product and 4 mg PCP/kg gave reason for serious concern. Analyses of samples collected to follow up these findings confirmed these high levels in certain batches; even higher levels were detected in few cases. However, also uncontaminated guar gum was found.

Legal limits

As regards the reference point of action for unacceptable levels of dioxins and pentachlorophenol in guar gum, the Commission services sent the following information to the competent authorities of the Member States in the interest of an uniform approach within the EU:

- Pentachlorophenol should be absent in guar gum (and also other food products). Regulation (EC) No 396/2005 [1] does not establish an MRL for pentachlorophenol. However it is foreseen in a draft Commission Regulation amending Regulation 396/2005 currently notified to WTO for comments that for pentachlorophenol the default MRL of 0.01 mg/kg (limit of quantification) would apply for all foods and feeds. Currently national MRLs exist of 0.01 mg/kg and 0.05 mg/kg. Therefore any quantified level of pentachlorophenol in guar gum is to be considered as unacceptable.

- As regards dioxins: No maximum levels have been established for dioxins in guar gum by Commission Regulation (EC) 1881/2006 [2]. However to determine what is to be considered as an unacceptable level, reference can be made to the maximum level existing for vegetable oils and fats with 0.75 pg WHO-PCDD/F-TEQ /g fat (which is in the case of pure vegetable oils and fats also 0.75 pg WHO-PCDD/F-TEQ /g product) or reference can also be made to the action level set by Commission Recommendation 2006/88/EC [3] for fruits vegetables and cereals which is 0.4 ng/kg product or 0.4 pg/g product. Following the requirement that contaminant levels shall be kept as low as can reasonably achieved by following good practices at all the stages of production, processing and distribution (Article 2 (2) of Council Regulation (EEC) 315/93 [4]), levels of dioxins (PCDD/F) in guar gum should be lower than 0.75 pg WHO-PCDD/F-TEQ /g product (or 0.75 ng PCDD/F- WHO-TEQ /kg product).

Levels higher than 0.75 pg WHO-PCDD/F-TEQ /g product are to be considered as unacceptably contaminated with dioxins

Results and discussion

The presence of dioxins is related to the presence of pentachlorophenol, as confirmed by so called "pentachlorophenol pattern" found by analysis of dioxins in contaminated guar gum. Therefore in order to gain time and money it appeared that the analysis of dioxin-like PCBs is not absolutely necessary as the presence of dioxin-like PCBs in relation to the dioxins and furans would be low, insofar it continues to be confirmed that the presence of dioxins is exclusively related to the presence of pentachlorophenol.

As the correlation factors between PCP and dioxin levels varied widely, the reliability of some results reported as part of the in-house quality control system of industry was deemed as questionable. The results of the 12 guar gum samples analyzed by private laboratories were compared with the results analyzed by the CRLs. In most cases the CRL for pesticides using single residue methods (CRL-SRM) found much higher PCP concentrations (more than a factor of 30 higher) compared to levels submitted by the Swiss company. For dioxin concentrations below 50 pg WHO-PCDD/F-TEQ/g the levels determined by CRL Freiburg and submitted by the Swiss company were mostly comparable, whereas for elevated concentrations above 50 pg WHO-PCDD/F-TEQ/g CRL Freiburg found considerably higher concentrations.

The considerable underestimations of the levels of PCP (possibly applied as Na-pentachlorphenol) can be the result of insufficient extraction due to wrong pH values: According to reports of CVUA Münster, Germany, a sample highly contaminated with dioxins (485 pg WHO-PCDD/F-TEQ/g) had a relatively low level of about 5 mg/kg PCP when directly extracted with an organic solvent (acetonitrile). However, extraction with acidified acetonitrile resulted in a 10fold increase to about 59 mg/kg PCP [5]. The CRL-SRM uses the QuEChERS-method (extraction with acetonitrile following buffering citrate salts at pH5; see http://www.crl-pesticides.eu/library/docs/srm/QuechersForGuarGum.pdf).

The reported results might be unreliable at the lower concentration ranges due to the fact that in the beginning of the incident, high levels of PCP and extremely high levels of dioxins were found, and industry / laboratories might have tried to make sure that guar gum samples with these high levels are quickly identified. However, the sensitivity might have been insufficient at the lower levels of the reference points of action for unacceptable levels set by the Commission (0.01 mg/kg PCP; 0.75 pg WHO-PCDD/F-TEQ/g). Reliable analyses in such a wide range of contamination (factors between minimum and maximum levels between 4000 and 15000) would require two different analyses: one screening method to detect samples in the range of high contamination (e.g. above 1 mg/kg PCP and above 10 pg WHO-PCDD/F-TEQ/g), then performance of another analysis in the range of the reference points of action for unacceptable levels.

The question was raised whether PCP analyses would be sufficient as screening method to make sure that guar gum samples do not exceed the level of 0.75 pg WHO-PCDD/F-TEQ/g product, if levels of pentachlorophenol in guar gum do not exceed 0.01 mg/kg. For evaluation of a possible correlation between PCP- and dioxin levels in guar gum, the Commission provided all data of analyses of guar gum batches, as available on 23 August 2007. The evaluation was updated with additional data sets of analyses of guar gum batches available on Sept. 12, 2007: A total of 765 data sets were provided; 155 samples thereof were analyzed for PCDD/F and PCP. The data was mainly derived from internal quality control of industry (analyses performed by private laboratories) and analyses of official laboratories.

The concentration ranges of PCP and dioxins found in the different batches of guar gum showed an extremely wide range of levels of PCP (from 0.0001 to 80 mg/kg) and dioxins (WHO-PCDD/F-TEQ: 0.06 – 738 pg/g).

Conclusions

From this study it can be concluded that guar gum (from this particular incident) containing a level of PCP below 0.01 mg/kg does not contain unacceptable levels of dioxins. Therefore, screening of guar gum samples for PCP allows to exclude samples with elevated levels of dioxins.

However, the evaluation of the above mentioned results for PCDD/F and PCP has shown that the reliability of the PCP results is questionable in some cases, especially in the low concentration range at the level of interest of 0.01 mg/kg for PCP. As a consequence, it is necessary to improve the reliability of methods for determination of PCP and dioxin concentrations at the level of interest. Therefore, in February 2008 the CRLs Freiburg and Stuttgart sent samples for a joint proficiency test “Determination of Dioxins and PCP in Guar Gum”. The evaluation

of the results of the PT will be presented and discussed at the next workshop of the CRL with NRLs for dioxins and PCBs in food and feed in July 2008.

References:

[1] Regulation (EC) No 396/2005 of the European Parliament and of the Council of 23 February 2005 on maximum residue levels of pesticides in or on food and feed of plant and animal origin and amending Council Directive 91/414/EEC (OJ L 70, 16.3.2005, p.1)

[2] Commission Regulation (EC) 1881/2006 of 19 December 2006 setting maximum levels for certain contaminants in food (OJ L 364, 20.12.2006, p. 5)

[3] Commission Recommendation 2006/88/EC of 6 February 2006 on the reduction of the presence of dioxins, furans and PCBs in feedingstuffs and foodstuffs (OJ L42, 14.2.2006, p. 26)

[4] Council Regulation (EEC) No 315/93 of 8 February 1993 laying down Community procedures for contaminants in food (OJ L 37, 13.2.1993, p. 1–3)

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