

PHTHALATE LEVELS IN BEVERAGES

Phthalates are very widely used as plasticisers in numerous polymer compounds and plastic materials. Some of these compounds are considered powerful endocrine disruptors, which recently led to a change in regulations governing their use in the fields of medicine and child care.



1 The regulation

In Europe, the use of phthalates in materials destined to be in contact with foodstuffs is subject to Commission Regulation (EU) No 10/2011 of 14 January 2011. This regulation pays special attention to certain phthalates listed in Annex IV of Commission Regulation (EU) no 143/2011 CE as reprotoxic (CMR category 1B) and foresees their pure and simple banning as of 1 January 2015. **These are benzyl butyl phthalate (BBP), dibutyl phthalate (DBP) and di(2ethyl hexyl)phthalate (DEHP)!** New regulations in Denmark preceded European and French ones, and came into effect at the end of 2012. French law no 2012-1442 of 24 December 2012 presently forbids the above phthalates in implantable medical devices. Di-n-octyl phthalate (DNOP), di-isononyl phthalate (DINP), and di-isodecyl phthalate (DIDP) were already forbidden for childcare articles by Directive 2005/84/EC and French decree 2006-1361 dated 9 November 2006.

There is no acceptable concentration in wines and spirits. At the present time, and at least until the beginning of the ban, Commission Regulation (EU) **No 10/2011 of 14 January 2011** is applicable, with the exception of any special tolerance for imports defined unilaterally by an EU member state. It is thus necessary to monitor the presence of the following molecules, especially those listed as most toxic (BBP, DBP and DEHP) in wines, spirits and the materials that come into contact with them.

2 Specific Migration Limits (SML) in alcoholic beverages - mg/kg

Molecule	Abbreviation	SML mg/kg
Benzyl butyl phthalate	BBP	30
Diethyl phthalate	DEP	Non-authorized (<0.01)
Dibutyl phthalate	DBP	0.3
Dimethyl phthalate	DMP	Non-authorized (<0.01)
Di-iso-methyl phthalate	DIMP	0.05
Diethylhexyl phthalate	DEHP	1.5
Di-n-octyl phthalate	DNOP	60
Di-iso-nonyl phthalate	DINP	Σ DINP + DIDP < 9
Di-isodecyl phthalate	DIDP	
Di-allyl phthalate	DAP	Non detected (=0.01)
Di-iso-pentyl phthalate	DIPP	Non-authorized (<0.01)
Di-iso-butyl phthalate	DIBP	Non-authorized (<0.01)

Regulation no. 10/2011 also includes a positive list, i.e. one of all authorised molecules in materials that come into contact with foodstuffs. Those molecules that are not cited are prohibited, and thus are not allowed in such materials.



3 Origin

Many materials, coatings and lubricants can be a source of phthalate pollution. Containers made of high density polyethylene (HDPE) or low density polyethylene terephthalate (PET) and polypropylene (PP) should not have significant quantities of phthalates. However, polyvinyl chloride (PVC) and its derivatives are a major source of contamination. Epoxy-based resins are also frequently a significant source of pollution. An increase in the alcoholic degree is accompanied by the increased risk of extracting phthalates. Therefore, it is advised to do migration testing in simulated environments to monitor the harmlessness of materials in contact with foodstuffs and to eliminate undesirable sources of pollution.

4 Taking samples

Use only previously rinsed glass bottles full of the beverage to analyse, and keep the stopper separate with aluminium foil.

Necessary volume for analysis = **100 ml**

5 Analysis

Analytical method: **SBSE-TD-GCMS**

Concentration of 12 phthalates **Deadline: 72 hours**

Light phthalates:

DMP-DEP-DBP-DIMP-BBP-DEHP-DIBP-DAP-DIPP

Limit of quantification: 0.01 mg/l

Heavy phthalates:

DINP-DIDP-DNOP

Limit of quantification: 0.5 mg/l

Test results interpreted in relation to Commission Regulation (EU) No. 10/2011 dated 14 January 2011.

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