

International Calibration Extract 3 (ICE-3)

Description

The ICE-3 is a hop extract containing a specified concentration of α - and β -acids. It replaces the ICE-2 as the international calibration extract hop standard of the European Brewery Convention (EBC) and the American Society of Brewing Chemists (ASBC).

Calibration

(Abstract of the Press Release «New International Calibration Standard (ICE-3) for HPLC Analysis of α - and β -acids»; Submitted on behalf of the International Hop Standards Committee by Martin Biendl, co-chairman).

In 2006 the Working Group of Hop Analysis (AHA) initiated the preparation of the replacement of the ICE-2. Similar efforts were undertaken as in case of the selection of ICE-2. In the end again a de-oiled supercritical CO₂-extract «Hallertau Perle» was selected as the most suitable replacement material. Following preparation, in November 2009 members of the International Hop Standards Committee (IHSC) carried out a collaborative trial in order to validate the new standard and assign values to the concentration of the α - and β -acids. This work included a collaborative HPLC study in which the prospective new standard was crosschecked against the existing standard.

As would be expected, the composition of ICE-3 is not identical to the standard it replaces, but it can be used in exactly the same way. No significant differences were observed when quantifying α -acids using ICE-3 vs. ICE-2. Such differences can be expected to be within the normal range of experimental error.

But users will find that the ICE-3 results for β -acids will differ significantly from ICE-2. The magnitude of this difference can be expected to be a relative drop of around 5% (i.e. the new standard gives a lower result).

However, the committee agreed that the use of ICE-3 will lead to a truer result than that obtained using ICE-2 as many efforts have now been undertaken to find a suitable primary standard for β -acids. For that purpose the dicyclohexylamine («DCHA») salt of β -acids was prepared. The purity of DCHA- β -acids could be ascertained by elemental analysis. For α -acids both «pure humulone» as well as o-phenylenediamine («OPDA») salts of α -acids served as primary standards. The results of the international collaborative trial obtained with these different types of primary standards were then compared to the results obtained with ICE-2 as external calibration standard. As in case of the α -acids there were no significant differences but almost identical results the International Hop Standards Committee (IHSC) agreed to assign the α -acids values of ICE-3 based on external calibration with ICE-2 whereas in case of β -acids the values were based on external calibration with dicyclohexylamine («DCHA») salt of β -acids

The following composition of ICE-3 has been determined accordingly and agreed on by the International Hop Standards Committee (IHSC):

Cohumulone	13.88 %	Colupulone	13.44 %
N+adhumulone	30.76 %	N+adlupulone	10.84 %
Total α -acids	44.64 %	Total β -acids	24.28 %

ICE-3 replaces ICE-2 and has been valid since September 1, 2010.

Use of the ICE-3

ICE-3 may be used as a reference or control for the following methods of analysis:

Analytica-EBC: Methods 7.7 and 7.8.

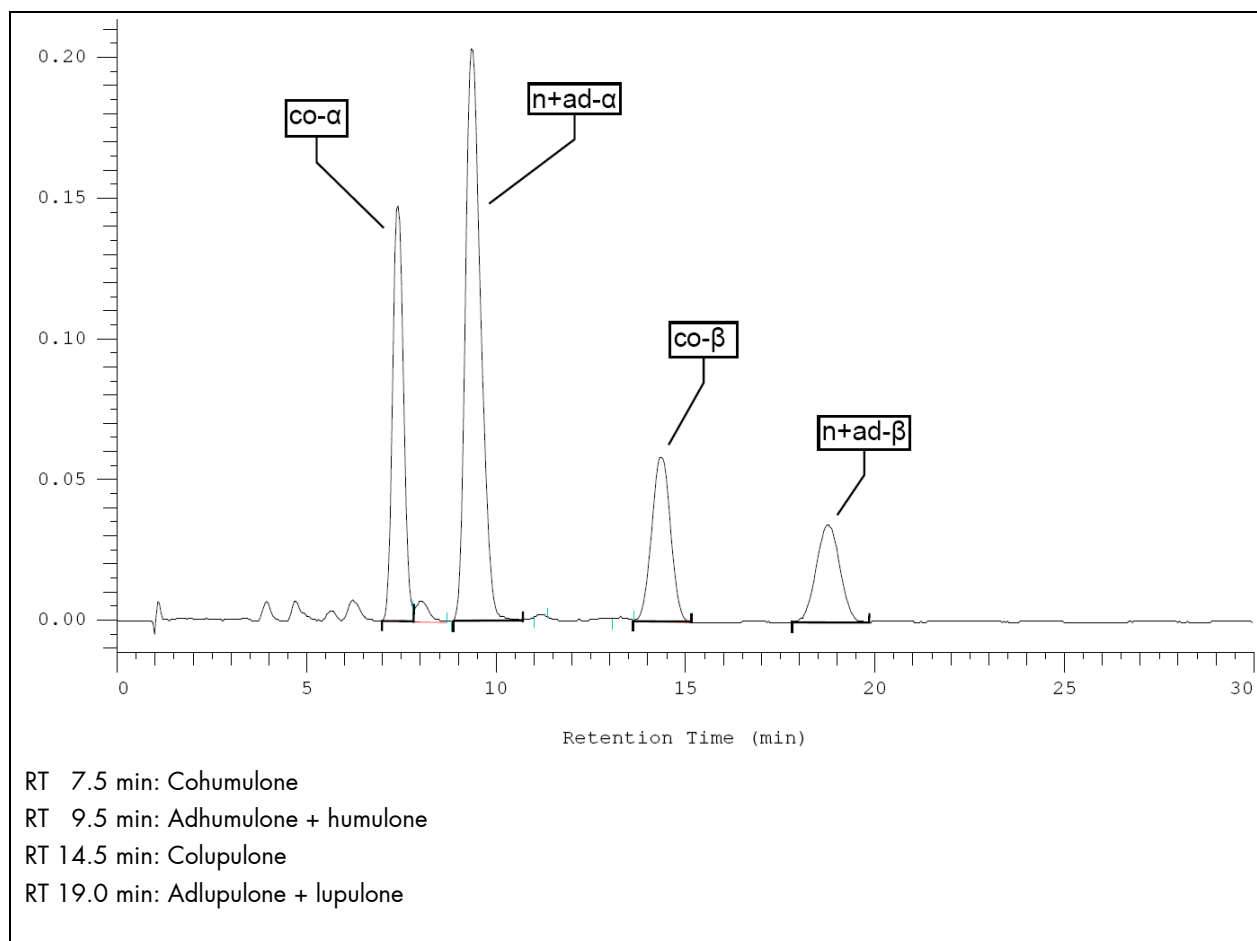
ASBC Methods of Analysis: Hops-14 and Hops-16.

Storage und Handling

Store the calibration extract, flushed with CO₂ or N₂ at -20 °C.

For use, heat up the frozen extract sample to about 20 °C and homogenize the entire content of the jar well by stirring it vigorously with a spatula. Prepare a calibration extract solution according to Analytica-EBC method 7.7 or ASBC Hops-14. To avoid frequent heating of the whole extract, the contents of the jar may be subdivided into smaller, single-use portions in the following manner: Heat up the frozen extract to about 20 °C and homogenize the entire content of the jar well by stirring it vigorously with a spatula. Distribute the whole calibration extract from the jar into individual glass vials as follows: weigh approximately 0.5 g of extract into individual glass vials and record weights to the nearest 0.1 mg. Flush the vials with CO₂ or N₂, close gas-tight and store in a freezer at -20 °C.

Reference HPLC-Chromatogram of ICE-3



September 1, 2010/Ld