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New International Calibration Standard (ICS-I3) for HPLC Analysis of Iso- α -acids

Submitted on behalf of the International Subcommittee for Isomerized Hop α -Acids Standards

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The International Subcommittee for Isomerized Hop α -Acids Standards (ISIHAS), announces the release of a new calibration standard, ICS-I3, for the HPLC analysis of iso- α -acids. This standard replaces ICS-I2, which is now withdrawn.

In April 2001, the ASBC, EBC, IoB (now IBD), and BCOJ approved the release of a set of HPLC standards for use in the quantitative determination of isomerized and reduced-isomerized α -acids in hop products and in beer. Four standards were produced:

- DCHA-Iso, **ICS-I1** (Iso- α -acids standard);
- DCHA-Rho, **ICS-R1** (Rho-iso- α -acids standard);
- Tetra, **ICS-T1** (Tetrahydroiso- α -acids standard);
- DCHA-Hexa, **ICS-H1** (Hexahydroiso- α -acids standard).

The purity of each standard was determined using various HPLC procedures, elemental analysis and other methods. In each case, the total content of major isomers and homologs was declared and, before release, the stability of the standard was assessed and recommendations made regarding storage and method of use. An isocratic version of EBC Method 7.8 (now EBC 7.9) was recommended as a convenient and generally applicable HPLC method for use in the analysis of unknown samples containing isomerized or reduced isomerized α -acids.

Over a period of 2½ years, the subcommittee monitored the stocks of the four standards via careful HPLC analysis, finally concluding that the standards had maintained their declared compositions and had not undergone significant chemical change.

Following exhaustion of the original stocks, the “Iso”, “Rho” and “Tetra” standards were replaced by new standards, ICS-I2, ICS-R2 and ICS-T2.

Release of ICS-I3

Stocks of DCHA-Iso (ICS-I2) are now almost exhausted. In anticipation of this, in 2007 the subcommittee initiated the preparation of a replacement standard. The new standard was prepared in the laboratories of Kalsec, Inc., under the guidance of subcommittee member, Louis Burroughs. Packaging was also carried out in the same laboratory. The subcommittee records its considerable appreciation to Louis Burroughs and his colleagues for all their skilled input to the successful manufacture of this new standard.

Following preparation, subcommittee members conducted extensive analysis in order to validate the new standard and assign a value to the content of “Iso” isomers, this work including a collaborative HPLC study in which the prospective new standard was crosschecked against the existing standard.

This new standard, ICS-I3, now becomes the recommended standard and should be used for commercial transactions as well as for quality control purposes.

Use of the new iso standard

As would be expected, the composition of ICS-I3 is not identical to the standard it replaces, but it can be used in exactly the same way. Dependent upon circumstances, users may find that the results for an unknown sample may differ slightly according to whether the old or the new standard has been used. In most cases though, the differences observed when quantifying iso- α -acids using ICS-I3 instead of ICS-I2 will be found to be very slight. The magnitude of this shift will vary according to the actual isomeric (cis- and trans-) and homological (co-, n- and ad-) composition of the unknown sample, but can be expected to be usually within the normal range of experimental error. ICS-I3 has a substantially lower ratio of co-isohumulone than its predecessor and better reflects the ratios found in commercially available Iso products.

Stocks of ICS-I3 are being divided between ASBC (in USA) and Labor Veritas (in Switzerland), from which sources analysts can purchase the new standard in the usual 250mg vials.* Orders are dispatched by express mail to minimize risk of damage in transit.

Detailed information pertaining to each standard, including full instructions for use, is available from ASBC or Labor Veritas on request and is automatically supplied with each purchase. ICS-I2 will now be available only while stocks last.

* Purchasers in the USA, Canada, Central and South America should contact ASBC headquarters (asbc@scisoc.org; tel: +1 (651) 454-7250), while those in Europe and Africa should direct enquiries to Labor Veritas, Zürich, Switzerland (admin@laborveritas.ch; tel: +41 (0) 44 283 29 30). Persons ordering from other parts of the world may make their approach to either party.