European Brewery Convention

International Subcommittee for Isomerized Hop α-Acids Standards

PRESS RELEASE

New International Calibration Standard (ICS-R2) for HPLC Analysis of Reduced (*rho*) iso-α-acids

(Submitted on behalf of the International Subcommittee for Isomerized Hop α-Acids Standards by Richard Wilson, Chairman).

The International Subcommittee for Isomerized Hop a-Acids Standards (ISIHAS), announces the release of a new calibration standard, ICS-R2, for the HPLC analysis of reduced (*rho*) iso-α-acids. This standard replaces ICS-R1, which is now withdrawn.

Background

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.

In 2004, following exhaustion of the original stocks, the “Iso” and “Tetra” standards were replaced by new standards, ICS-I2 and ICS-T2.

Release of ICS-R2

Stocks of DCHA-Rho (ICS-R1) are now almost exhausted. In anticipation of this, in 2004 the subcommittee initiated the preparation of a replacement standard. As for ICS-I2 and ICS-T2, the new standard was prepared in the laboratories of John I Haas, Inc., in this instance by Jianping Xu under the guidance of subcommittee member, John Paul Maye. Packaging was also carried out in the same laboratory. The subcommittee records its considerable appreciation to Mr Xu, Dr Maye and their other co-workers 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 "Rho" isomers, this work including a collaborative HPLC study in which the prospective new standard was crosschecked against the existing standard.

This new standard, ICS-R2, now becomes the recommended standard and should be used for commercial transactions as well as for quality control purposes.

Use of the new Rho standard

As would be expected, the composition of ICS-R2 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 *rho*-iso-α-acids using ICS-R2 instead of ICS-R1 will be found to be very slight and usually within the normal range of experimental error. However, users should note that, following evaluation of LC-MS and NMR data obtained during examination of a sample of a commercial Rho product by Nils Nyberg and Steen Bech Sørensen at the Carlsberg Research Center, and subsequent LC-MS examination of ICS-R2 and some commercially available Rho products by Doug Williams and Lou Burroughs at Kalsec, Inc., the committee has also decided to recommend a concurrent change to the way in which the total Rho content of an unknown sample should be determined. This change will tend to increase the measured "Rho" content of an unknown sample of a commercial Rho product (or of the worts or beers prepared from it), typically by about 3 - 6 %. Hence products that are purchased on a UV Spectrometric basis will be found to be more concentrated than hitherto when analyzed by HPLC. Details of the procedure to be used are given in literature that accompanies the standard.

Rho products that are sold and purchased on the basis of HPLC analysis against ICS-R2, should now be assumed to have been analyzed according the newly recommended procedure.

General revision of literature

Concurrently with the release of ICS-R2, the committee has approved the revision of some other items of literature that are supplied with each purchase of an ICS series standard of any type. As well as the specific change to the way in which samples containing *rho*-iso-α-acids should be assessed, users are also advised to take particular note of alterations to the User's Guide to the International Calibration Standards that incorporates the recommended method of analysis. (Changes to this document apply to all the standards and are intended merely to assist improvement of the accuracy of analysis - they will not otherwise alter the result).

How to purchase

Stocks of ICS-R2 are being divided between ASBC (in USA) and Labor Veritas (in Switzerland), from which sources analysts can purchase the new standard in the usual 250 mg 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-R1 will now be available only while stocks last.

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

RJHW 4th November 2005