

Pre-isomerised hop products – potential and practical use

An up-to-date overview

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Iso-alpha-acids are formed from alpha-acids contained in hops (Fig. 1). Since 1947 when this transformation was explained chemically by Rigby, there have been efforts to have the reaction proceed under optimised conditions. When isomerising alpha-acids during wort boiling, losses of about 50% arise.

In the first instance, pre-isomerised hop products have therefore been developed mainly in order to achieve a more efficient i.e. a more cost-effective utilisation of hops. In addition, there are possibilities available nowadays whereby iso-alpha-acids are subjected to further modification by a chemical hydrogenation reaction. These are also classed as pre-isomerised hop products. One distinguishes here between rho-iso-alpha-acids which contribute to light stability, and tetrahydro- and hexahydro-iso-alpha-acids (Fig. 2) which improve both light stability and head retention of beer. The light stability achieved by reduced iso-alpha-acids is regarded as a prerequisite for filling of beer in clear bottles; this has possible marketing implications.

As all pre-isomerised hop products differ from conventional products in terms of chemical composition; differences in flavour impression can arise in the finished product. They are therefore of potential interest, among other things, when the objective is to develop new beer brands. The extent of the shift in flavour is dependent on numerous factors, e.g. on the type of product, the type of beer or the brewing process used in production.

Some pre-isomerised products are added to the wort in exactly the same way as conventional pellets or extracts, others are

The classical means (and only means allowed by the Purity Law) of bittering beer is by addition of hops or conventional hop products (pellets, extracts) to boiling wort. Iso-alpha-acids arise during boiling, these are the main bittering substances in beer. Should iso-alpha-acids be formed not in boiling wort but external to the brewery, one speaks of pre-isomerised hop products.

dosed only into the “beer stream” ahead of or during filtration. This clearly improves yield still more as losses during fermentation are avoided. This group taken together are classed as “downstream products”.

Pre-isomerised hop products can thus be classified as follows:

- Pre-isomerised products for addition to wort
 - iso-pellets
 - IKE
 - PIKE
- Pre-isomerised products for addition after fermentation (downstream products)
 - Iso
 - Rho
 - Tetra
 - Hexa

The individual product types will be described below and discussed with reference to recent publications.

■ Pre-isomerised pellets for adding to wort (“iso-pellets”)

Iso-pellets are produced similar to type 90 pellets. Prior to pelletising, one mixes a catalyst, e.g. magnesium oxide, into the

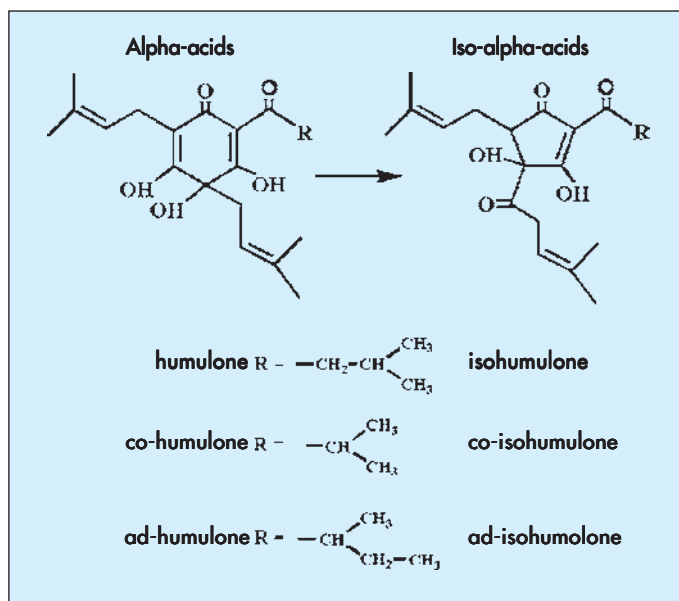


Fig. 1
Isomerisation of alpha-acids

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Parameter	Starting hops	Iso-pellet
% alpha-acids	No data	9.8
% iso-alpha-acids	No data	0.1
% beta-acids	No data	2.8
Total oil (mg/100 g), of which % relative in:	0.6	1.0
Myrcene	9.2	47.0
Caryophyllene	10.3	7.9
Humulene	24.9	18.1

hop powder. Pelletising and packing are carried out in the same way as for conventional pellets. Instead of cold storage, the packed pellets are initially stored at 50°C for 8 – 12 days. Under these conditions, alpha-acids are isomerised. The reaction is analytically monitored. After isomerisation is complete, the goods are cold stored just like conventional pellets.

As reported by *Taylor et al. (1)*, the bitter substance fraction, with the exception of an alpha-acid isomerisation, remains practically unchanged in producing iso-pellets (“there is little evidence of any deterioration of the resins such as oxidised alpha-acids”). Hop oil content may, however, drop whereby the highly volatile myrcene is reduced to a considerably more pronounced extent than the heavier compounds, caryophyllene and humulene (Table 1).

In their paper, *Taylor et al. (1)* provide a detailed description of commercial-scale use of iso-pellets. It was found that, when using iso-pellets, an iso-alpha-acid yield of 65% could be achieved in the finished beer whereas comparative brews using conventional pellets achieved a maximum yield of 37%. It was also shown that yield levels achieved with iso-pellets were largely independent of the time of addition. Addition could take place up to 10 minutes prior to the end of wort boiling. The use of aroma hops in the form of iso-pellets for producing hop aroma beers of the “cask-conditioned ale” type is also possible and is discussed. For this particular application, hop savings are particularly large because, in comparison to conventional aroma hop addition, losses of non-isomerised alpha-acids are avoided. When developing this approach to the full, this way of hopping can create innovative products (“interesting and exciting new product and novel flavour profile opportunities”), when e.g. complete addition in the form of aroma hop iso-pellets takes place shortly before the end of wort boiling.

■ Pre-isomerised extracts for adding to wort (“IKE”, “PIKE”)

Iso-extracts produced by isomerising conventional carbon dioxide extracts using a catalyst are an alternative to iso-pel-

lets and can also be added during wort boiling.

Production takes place by mixing the carbon dioxide extract with water and catalyst (e.g. also magnesium oxide) and heating to about 90°C. Subsequently, the catalyst is removed again by acidification. The resulting product is known in the trade as “IKE” (isomerised kettle extract). “PIKE” (potassium-form isomerised kettle extract) is another version where water and potassium ions are subsequently added in order to transform the free iso-alpha-acids into a potassium salt form which is more readily soluble in wort. Due to its good solubility, “PIKE” is especially suitable for last-minute hop additions. Examples of compositions of “IKE” and “PIKE” are shown in Table 2.

At the World Brewing Congress (2000), a paper was read on the subject of production and use of these products (2). As reported by *Wilson*, brew tests on a pilot scale showed that conventional extracts can be substituted by “IKE” and “PIKE” without giving rise to significant changes in flavour (“without apparent change to the beer flavour”).

In the tests described by *Wilson*, the yields achievable with these products were around 70% (iso-alpha-acids in beer relative to added quantities of iso-alpha-acids). The highest yield achieved (75.7%) was observed when adding “PIKE” after the end of boiling, immediately prior to a whirlpool rest of 30 min.

Table 2 Composition of a carbon dioxide extract (variety: Chinook) and the “IKE” (isomerised kettle extract) and “PIKE” (potassium-form isomerised kettle extract) products made from it according to *Wilson et al. (2)*

Parameter	Carbon dioxide extract	“IKE”	“PIKE”
% alpha-acids	55.9	0.1	0.2
% iso-alpha-acids	0.0	56.8	47.4
% beta-acids	17.6	16.4	13.7
Total oil (mg/100g), of which % relative in:	9.0	8.6	8.7
Myrcene	23.7	17.4	24.5
Caryophyllene	12.8	10.5	11.2
Humulene	26.1	25.4	24.2

Table 1 Composition of iso-pellets compared to starting hops (variety: Target) according to *Taylor et al. (1)*

■ Pre-isomerised extracts for use after fermentation (“Iso”)

“IKE” and “PIKE” contain a series of other hop components (beta-acids, non-specific resins, hop waxes, hop oils) apart from iso-alpha-acids. Only when these fractions are completely removed is it possible to make the addition after fermentation.

In producing such a product, a conventional extract is first isomerised using a suitable process. Subsequently, hop oils, waxes, non-specific resins and, after a pH shift, beta-acids and remainders of non-isomerised alpha-acids are separated so as to leave an aqueous solution of iso-alpha-acids. After a further pH reduction, the free iso-alpha-acids are precipitated and separated out from the aqueous phase. They are usually stored in this form up to the final adjustment step. Just prior to delivery to the brewery, iso-alpha-acids are transformed into a potassium salt form and adjusted with demineralised water to a sales concentration of 20% (“Iso 20%”) or 30% (“Iso 30%”). This aqueous potassium isohumulate solution is then filled into drums.

Such “Iso”-products can be used at various points in the brewing process. Basically, the later the point at which addition takes place, the better is the yield factor. It can go up to 90%.

But in order to assure normal wort boiling and have fermentation proceed without upset while avoiding microbiological problems, only a part of conventional hop addition is oftentimes substituted by “Iso” in the brewing process. Frequently, the “Iso” portion is 30 – 40%. Consequently, some breweries use the “beta-fraction” (also referred to as “base extract”) separated during “Iso” production and add this during wort boiling. In some cases, “Iso” is added only for fine adjustment/correction of bitterness which conventionally hopped green beers (or those hopped with isomerised products during wort boiling) exhibit after fermentation. This procedure is used i.a. for high-gravity brewing processes.

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no) and then subjected to the effects of sodium borohydride or hydrogen gas in the presence of a catalyst (e.g. palladium on activated carbon) (Fig. 2). After the reaction has gone to completion, unused reagents and catalyst are separated by filtration and the solvent driven off under vacuum. The reduced iso-alpha-acids are then dissolved in diluted potassium hydroxide solution and adjusted to the desired final concentration. The corresponding products are referred to simply as "Rho", "Tetra" or "Hexa". In the case of "Rho", the final concentration is very often 35 % rho-iso-alpha-acid, "Tetra" mostly contains 10 % tetrahydro-iso-alpha-acids. In the case of "Hexa", glycols are usually used as solubilisers for stabilising the aqueous solution of hexahydro-iso-alpha-acids.

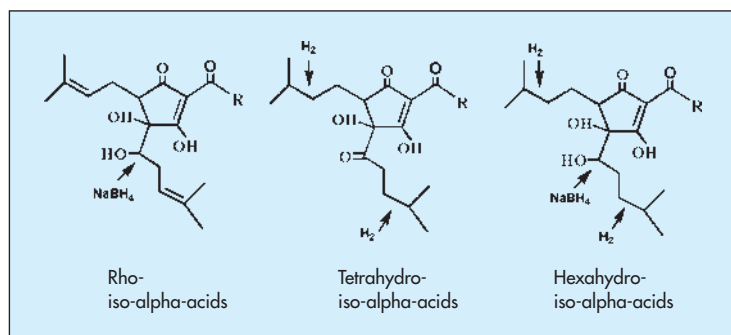


Fig. 2 Reduced iso-alpha-acids (chemical action location marked)

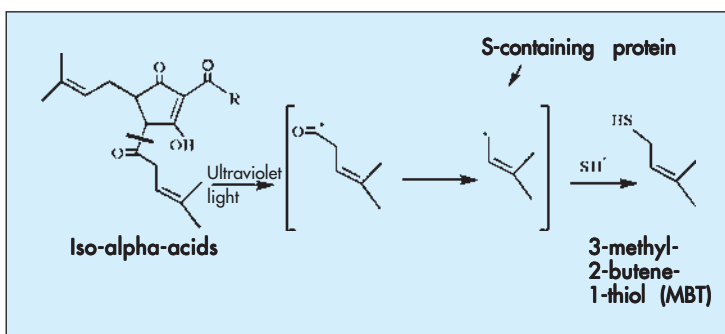


Fig. 3 Development of lightstruck flavour

On occasion, a reduced taste stability is reported to be associated with "Iso", this is attributed to unsaturated fatty acids present in trace quantities. Foster et al. (3) confirm in their publication that the concentration of stale-flavour carbonyls increases in beer in keeping with the levels of unsaturated fatty acids present in the "Iso" product. They recommend that breweries who have noticed a negative influence on taste stability when using "Iso" add the product prior to fermentation as the yeast can metabolise fatty acids and thus removes them. A reduced yield has, however, to be accepted.

Pre-isomerised reduced products for use after fermentation ("Rho", "Tetra", "Hexa")

Development of this product family began after Kuroiwa and Hashimoto established in 1961 that iso-alpha-acids play a key role in creating a lightstruck flavour. Light in the near ultraviolet spectrum gives rise to a dimethylallyl radical by cleaving off a side chain of the iso-alpha-acids. This can lead to formation of a foul-smelling compound 3-methyl-2-butene-1-thiol (MBT) when it reacts with a sulphhydryl group (Fig. 3). This reaction is regarded as the cause for the lightstruck flavour in beer.

The taste threshold value of MBT is extremely low, under 10 ppt.

This reaction cannot take place when the corresponding side chain of the iso-alpha-acid is reduced. A process was patented for the first time in 1962 which, by reduction of the carbonyl group, gave rise to the so-called rho-iso-alpha-acids (sometimes also referred to as dihydro-iso-alpha-acids). These cannot form MBT.

As even traces of non-reduced iso-alpha-acids can still cause a lightstruck flavour, a patent was granted about 10 years later, describing production of reduced iso-alpha-acids from beta-acids. So-called tetrahydro- and hexahydro iso-alpha-acids arise in this case. The discovery of a foam-stabilising effect then led to additional novel applications for these compounds. Meantime, processes for production of tetrahydro- and hexahydro-iso-alpha-acids from alpha-acids or iso-alpha-acids have been developed.

In commercial situations, iso-extracts for example can serve as starting products for production of all forms of reduced iso-alpha-acids. By acidification, free iso-alpha-acids are formed initially, these are subsequently dissolved in a solvent (e.g. etha-

The use of reduced iso-alpha-acids served in the first instance to achieve light stability of beer. The relatively non-polar tetrahydro- and hexahydro-iso-alpha-acids are also very efficient foam stabilisers. They have been increasingly used for this purpose and replace other additives (e.g. alginates).

Bittering capacity of reduced iso-alpha-acids

The bittering capacity of various reduced iso-alpha-acids compared to iso-alpha-acids is discussed frequently. In some instances, the literature contains divergent opinions. This is due to differences in the test set-ups which differ from one to the next or to inadequately defined or non-uniform analysis.

In a recent project at the Faculty of Technology for Brewery I at the Technical University of Munich-Weihenstephan (4), the bittering capacity of i.a. rho- and tetrahydro-iso-alpha-acids was investigated in detail. In the test set-up, various quantities of "Rho" or "Tetra" were added to unhopped beers. A beer conventionally hopped with carbon dioxide extract was used as a reference. All beers were taken from the same first wort. The resulting concentrations of iso- or reduced iso-alpha-acids were measured specifically using HPLC. Subsequently, the bitter units of the beers were estimated by a large taster panel (35 people). The results are shown in Table 3. The "perceived bitterness" of tetrahydro-iso-alpha-acids was just about at the same level as that of iso-alpha-acids, whereas rho-iso-alpha-acids were found to be less bitter.

This investigation has produced results which are in good agreement with those of Seldeslachts et al. (5) presented in a poster at the EBC Congress in Cannes (1999). They also made an attempt to relate the "perceived bitterness" in beer via the "tasted

	Perceived bitterness in beer per mg/l	
	Back et al. (4): "Estimated units"	Seldeslachts et al. (5): „Tasted bitter bnits“
Iso-alpha-acids	1.0	1.0
Rho-iso-alpha-acids	0.6 – 0.7	0.47
Tetrahydro-iso-alpha-acids	1.0 – 1.1	1.05

Table 3 Perceived bitterness in beer from reduced iso-alpha-acids compared to iso-alpha-acids at identical concentrations in mg/l (measured by HPLC)

bitter units" to the concentrations specifically measured using HPLC analysis. The corresponding beers also contained different concentrations of the particular reduced iso-alpha-acids.

Both of these investigations referred to above reported on the bittering capacity determined in beer, whereas other authors measured bitterness in water (6). Here, "tetrahydro-iso-alpha-acids" were found to be more bitter by a factor of 1.9 and rho-iso-alpha-acids were found to be less bitter by a factor of 0.6 compared to iso-alpha-acids. Bitterness arising from hexahydro-iso-alpha-acids was reported to be approximately the same (factor 1.1). The difference in bittering capacity, in particular in the case of tetrahydro-iso-alpha-acids in the "beer" matrix compared to pure water, is apparently related to masking factors. It may be expected that the degree of difference in bitterness will fluctuate from one beer type to another. The exact bittering capacity of reduced iso-alpha-acids therefore has to be specifically determined in each brewery.

Many results published in the literature are unfortunately not based on concentrations measured by HPLC but rather on spectrophotometric measurements of bit-

ter units (Analytica-EBC 9.8 method). Due to a modified molecular structure, reduced iso-alpha-acids show extinction coefficients different from iso-alpha-acids at a measurement wavelength of 275 nm so that the corresponding results are not directly comparable one to another. *Jorge and Trugo (7)* report on conversion factors which may serve to provide an objective comparative basis (see also Table 4).

■ Analysis of pre-isomerised hop products

The work of *Jorge and Trugo (7)* discusses yet another interesting and important aspect which has to be taken into account in quality control when using pre-isomerised hop products in the brewery.

In determining bitter units in accordance with the Analytica-EBC 9.8 method, it is well known that the measured absorption at wavelength 275 nm is multiplied by a factor of "50". Strictly speaking, this formula applies only when bitterness comes from hops or conventional hop products. Beta-acids and non-specific resins as well as compounds formed from alpha-acids not converted to iso-alpha-acids contained in these products also make a contribution to beer bitterness. The literature refers to

these compounds as "non-iso-alpha-acids" bittering compounds" (NIBC). The contribution of these NIBC is reflected in the "50" factor in the BU determination.

Should pre-isomerised products be used, various amounts of NIBC are present depending on the type of product. In iso-pellets, "IKE" and "PIKE", beta-acids and non-specific resins are still present. The alpha-acids are, however, completely isomerised whereas in the case of conventional products, about half are converted to NIBC. NIBC are less bitter than iso-alpha-acids. When using isomerised products, the factor should be correspondingly increased in determining bitter units. It is difficult to hit the factor exactly because, in practice, it depends on beer and brewery-specific conditions. *Taylor et al. (1)*, *Wilson et al. (2)*, and *Jorge and Trugo (7)* recommend factors of "55 – 58" in their papers.

The situation becomes more extreme when downstream products are used, i.e. when no NIBC whatsoever are present any longer. In the case of "Iso", *Jorge and Trugo (7)* suggest a factor of "70.2". In addition, when using "Rho", "Tetra" or "Hexa", the differing adsorption of reduced iso-alpha-acids at 275 nm has to be taken into ac-

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neously hopping during wort boiling (on microbiological or flavour grounds). The light stable isomerised extract (light stable kettle extract "LIKE") is produced by converting "IKE" with sodium borohydride and subsequently separating the catalyst. Compared to "IKE", only the iso-alpha-acids are substituted by rho-iso-alpha-acids in "LIKE". According to Wilson (2), first pilot tests with "LIKE" seem to be promising.

The downstream products "Iso", "Rho", "Tetra" and "Hexa" can be added after fermentation because, apart from the particular iso-alpha-acid type, they do not contain any other components. In order to achieve high yield figures, it is recommended that addition takes place as late as possible.

Continuous addition prior to or during filtration using a dosing pump is optimal. Dosing after filtration is not to be recommended because components which are incompletely dissolved may give rise to hazes in beer. Fig. 4 shows an example of dosage of "Tetra" in a commercial situation. It is important that addition takes place directly proportional to the beer flow rate.

As described by Seldeslachts et al. (5) in the above-referenced poster session presented at the EBC Congress in Cannes (1999), a reproducible addition of tetrahydro-iso-alpha-acids (measured in mg/l by HPLC) during beer filtration is achievable with suitable software in which the addition of "Tetra" through a mass flow meter is calculated in relation to the actual flow rate of beer in accordance with the following formula:

Addition:

$$\frac{\text{Target concentration in beer} \times \text{actual beer flow rate}}{\text{HPLC content "Tetra"} \times \text{yield factor}}$$

Units:

Addition	g/h
Target concentration in beer	mg/l
Actual beer flow rate	hl/h
HPLC content Tetra	g/100g
Yield factor	%

Should for example 4.0 mg/l of tetrahydro-iso-alpha-acids be the target concentration, with an actual beer flow rate of 500 hl/h, at a measured HPLC level of 9.1 g/100g and a (predetermined) yield of 85 %, it is necessary to add 2.586 g of "Tetra" per hour.

For some downstream products (in particular for "Rho"), considerable levels of precipitation have been observed at ambient temperature. In this case, it is necessary to heat up to 40 °C – 60 °C before addition so as to achieve homogeneous clear solutions.

Table 4 Specific adsorption coefficients and factors for determining bitter units (BU) in accordance with the EBC 9.8 method according to Jorge and Trugo (7)

	Extinction E (1 cm, 1%)	Factor for BU determination
Iso-alpha-acids	285	70.2
Rho-iso-alpha-acids	276	72.5
Tetrahydro-iso-alpha-acids	275	72.7
Hexahydro-iso-alpha-acids	245	96.1

count in comparing them with iso-alpha-acids. Table 4 provides a corresponding overview.

If these corrections in BU determination are made, quality control of beers produced with pre-isomerised hop products can be carried out as before in accordance with the Analytica-EBC 9.8 method.

HPLC analysis provides an alternative. Quite recently, suitable internationally recognised calibration standards have become available which can be used to test the hop product in question as well as for analysis of beer. An accurate yield measurement and determination of the economics is thus possible. The new standards were presented for the first time at the EBC Congress in Budapest (2001) (8). In Europe, they can be ordered as EBC calibration standards at the Veritas Laboratory (admin@laborveritas.ch). In the US, they are available via ASBC.

Methods for determining reduced iso-alpha-acids in beer have been published by Burroughs and Williams (9) as well as Harms and Nitzsche (10).

■ Addition of pre-isomerised products in commercial operations

Iso-pellets, "IKE" and "PIKE" can be added only during wort boiling because, apart from iso-alpha-acids, they contain a whole series of other hop components which have to be converted, driven off or separated out during wort boiling. In part, they are

also eliminated by yeast adsorption or by krausen separation during fermentation. They can be used in the same way as conventional products. The same automatic dosing equipment as is used for pellets or extracts is also suitable, if available.

The achievable yields of about 70% (iso-alpha-acids in the finished beer relative to the quantities of iso-alpha-acids added) are largely independent of the point in time at which they are added. Pre-isomerised products can also be added just prior to the end of boiling. This makes it possible to achieve better economics in aroma hop addition, and this may give rise to novel beer types.

Every brewery has to determine for itself whether the same flavour and the same aroma are achievable with isomerised products added during wort boiling as can be obtained with conventional products. If the objective is to substitute conventional products without changes in flavour, it is recommended that a careful stepwise approach be adopted in the context of a well planned testing programme.

Basically, downstream products which have been purified further can be added during wort boiling. Particularly in the case of "Rho", this variant is used sometimes in commercial situations in order to avoid microbiological problems.

At the World Brewing Congress, Wilson (2) even presented a novel product for production of light stable beers while simulta-

Table 5 Composition of downstream products compared to hops

Substance group	Hops	"Iso"	"Rho"	"Tetra"	"Hexa"
Bitter substances	Non-specific resins	-	-	-	-
	Alpha-acids	Iso-alpha-acids	Rho-iso-alpha-acids	Tetrahydro-iso-alpha-acids	Hexahydro-iso-alpha-acids
	Beta-acids	-	-	-	-
Essential oils	Myrcene, Caryophyllene, Humulene etc.	-	-	-	-
Polyphenols	Anthocyanogens, prenylflavonoids (xanthohumol)	-	-	-	-

Table 6 Reasons for using various downstream products

Improvement in ...	“Iso”	“Rho”	“Tetra”	“Hexa”
Utilisation of bitter substances	X		X	
Light stability		X	X	X
Head retention			X	X

In order to achieve good mixing with the beer, it is advantageous to dilute downstream products prior to usage with demineralised water to about 1 – 3 % solutions. In some instances, the use of potassium hydroxide is recommended to achieve an alkaline pH (e.g. 11). The corresponding manufacturer’s instructions have to be followed exactly.

When used under optimal conditions, yields of practically 90 % can be achieved with “Iso”. Due to inferior solubility in beer, somewhat lower rates of conversion are achieved with reduced downstream products as a rule.

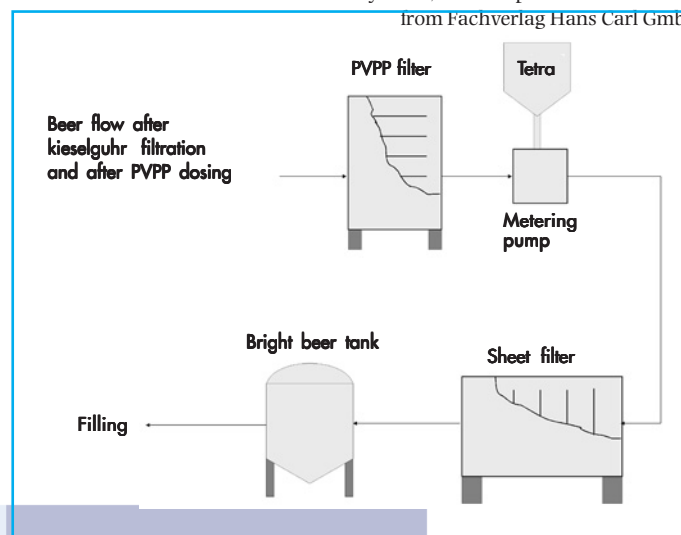
When reduced iso-alpha-acids (“Rho”, “Tetra” and “Hexa”) are used for light stability, it is necessary to be very careful that no alpha- or iso-alpha-acids whatsoever get into the wort or beer. All products must be completely free of these. Should a “beta-fraction” be added to wort boiling, this should also contain no residues of alpha-acids. Even yeast which has been recovered from a brew with conventional hop addition could carry over quantities of iso-alpha-acids which are sufficient to create a lightstruck flavour.

“Tetra” is used primarily for foam stabilisation. Experience has shown that concentrations of tetrahydro-iso-alpha-acids in beer should not exceed 7 ppm as this would give rise to an unnatural (“artificial”) foam. Hexahydro-iso-alpha-acids are usually used in even lower concentrations. “Hexa” in beer may tend to lead to haze problems.

In some instances, a mixture of tetrahydro- and hexahydro-iso-alpha-acids is used for foam stabilisation. This is based on the theory that various types of hydrogenated iso-alpha-acids can form different complexes with proteins and thus may improve head retention further.

In terms of flavour, beers produced with conventional hop products and with downstream products necessarily differ from one another. Only a specific iso-alpha-acid type is contained in the downstream products in comparison to the natural diversity of aroma substances. In contrast to a balanced and rounded bitterness, this gives rise oftentimes to a bitterness which

Fig. 4
Addition of “Tetra”
in commercial
operation
(example)



is regarded as “uniform”. In addition, all downstream products are completely free of aroma substances (essential oils) and polyphenols (Table 5).

The reasons for using the various types of downstream products are summarised in Table 6.

■ Summary

Pre-isomerised hop products are basically classifiable into two types. Products which are added during wort boiling (iso-pellets, isomerised kettle extract, potassium-form isomerised kettle extract) like conventional extracts or pellets achieve increases in yield of maximum 40 % to about 70 %, measured in the finished beer.

Downstream products (“Iso”, “Rho”, “Tetra”, “Hexa”) which can be added at a later point in time are even more economical. With optimised addition proportional to the beer flow rate prior to or during filtration, conversion rates of up to 90 % are possible depending on product type. Reduced pre-isomerised downstream products are mainly used for light stability or foam stabilisation.

In terms of flavour, beers produced with conventional hop products and with downstream products necessarily differ from one another. Only a specific (sometimes chemically modified) iso-alpha-acid type is contained in the latter in comparison to the natural diversity of bittering and aroma substances. This paper describes the various products and their application in commercial operations as well as analytical aspects drawing on recent publications.

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