Hop Aroma Component Profile and the Aroma Unit

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ABSTRACT

More than 250 essential oil components have been identified in hops by capillary gas chromatography. The hop aroma component profile (HACP), quantitatively measured as nanoliters per gram of hops (1 ppm, v/w), is composed of 22 of these hop oil compounds that have been reported to affect hop aroma. Similar steam distillation methods were used to measure the HACP of hops, wort, and beer. HACP analysis of seven commercial hop pellet samples (Cascade, Chinook, Cluster, Hallertau, Saaz, Tettnang, and Willamette) were made. It was found that within one variety, the total HACP could vary by as much as 50%. Subsequently, the hop aroma unit, which consists of 1 nl/g (1 ppm, v/w), the sum of the 22 hop oil constituents, was conceived. The hop aroma unit can be used as a standard measure of hop aroma content, just as the bitterness unit is used to measure bitterness.

Keywords: Aroma, Hop, Hop oil

Quantitatively, hops are a minor ingredient in brewing. Nevertheless, the contribution of hops to the flavor and aroma of beer is important. The relationship between the amount of α-acids in hops and bitterness in beer was elucidated many years ago. During the boiling of wort, α-acids are converted to bitter iso-α-acids. The measurement of bitterness units (BU) in beer is a test used worldwide, even though it is not specific for iso-α-acids (1). A number of factors affect the utilization of α-acids, but generally, the higher the α-acids content in hops, the fewer hops it takes to achieve a certain bitterness level. Kettle or "bittering" hops are added at the beginning of wort boil to achieve maximum use of α-acids, or preisomerized extracts are added after fermentation. Because each brewery has different utilization rates of α-acids to iso-α-acids (11), each brewery adjusts hopping rates based on the α-acid contents of the hops they use and the BUs specified for their product.

Research on the hop oil compounds with capillary gas chromatography (GC) has led to the identification of 250 compounds in the essential oil of hops. A number of these compounds, i.e., humulene oxidation products, have been isolated or synthesized and used in flavor evaluations (13,14,21,24). In addition, a great deal of work has been done on the detection of hop oil constituents in beer (4,10,16,18,20,23). Unfortunately, no consensus has been reached on which constituents contribute the most to a "hoppy" aroma in beer. Murray et al suggested that the proportion of alcohols to ketones influences the nature of late-hop flavor (19). They also have found varietal differences in beers brewed with single hop varieties (7-9). In England, the practice of dry hopping has led to the postfermentation addition of hop oil isolated from liquid carbon dioxide extracts (6). The amounts and composition of such extracts can be controlled fairly easily (27). Dry hopping seldom is done in the United States; instead, "aroma" hops generally are added just before the end of wort boil. The late addition of aroma hops is based on the fact that a considerable loss of hop aroma occurs during wort boiling because of the steam distillation of volatiles. Shorter boiling times increase the amount of hop oil constituents in the wort.

The major constituents of hop essential oil are monoterpenes and sesquiterpenes, and the proportions of these compounds are valuable in varietal identification (12,28), although they seldom are detected in beer (16,17). Oxygenated compounds present in hops have been detected in beers (22,24,29), and fermentation products from hop oil constituents also have been identified (20,26). Foster and Nickerson proposed measuring the amounts of 19 hop oil compounds per gram of α-acid as an expression of the potential aroma contribution and called this "Sigma" (5). The compounds were divided into three groups, excluding major hydrocarbons, consisting of floral-estery compounds, piney-citrus compounds, and humulene and caryophyllene oxidation products. The Sigma measurement presumes that the hopping rates will be adjusted according to α-acids content and that the aroma deriving from the hop oil constituents will vary depending on variety and storage. In this article, we propose that the required volume of hop oil constituents per gram of hops should be used as the criterion for adjusting the hopping rates of aroma hops and that the α-acids content should then be a secondary consideration. The list of hop oil compounds that comprise Sigma was modified to include the 22 compounds listed in Table I as the basis for a hop aroma component profile (HACP). Instead of cadinene(s), the individual compounds are given—α-muurolene, β-selinene, and Δ- and γ-cadinene. Nerolidol was deleted because it is difficult to detect in hop oil and is not really an oxidation product of humulene or caryophyllene. In Table I, the compounds in parentheses usually are not detected in fresh hop oil, at least under our experimental conditions. One aroma unit (AU) is defined as 1 ppm (nanoliters of oil per gram of hops or microliters per liter of wort and beer) of the sum of these compounds. This is analogous to the BU measurement in beer.

This article emphasizes the analytical procedures used to measure hop oil constituents in hops, wort, and beer and the effects of maturation, storage, and variety on the HACP. A subsequent article relates the application of these measurements to brewing (30).

EXPERIMENTAL

Hops and Hop Products

Fresh-picked green hops, dried and stored samples from the Oregon State University (OSU) world hop collection, and commercially prepared hop pellets all were analyzed. Hops usually are analyzed after they have undergone considerable processing (picking, dying, baling, and perhaps pelletizing or extraction). The analysis of hand-picked green samples provides information on the effects of picking, drying, and baling on the hop oil composition. Hop cones were hand-picked from sidearms collected from commercial hop yards. The green samples were analyzed for dry matter, oil, and resin content and composition.

TABLE I
Hop Aroma Component Profile Constituents

Humulene and Caryophyllene Oxidation Products	Floral-Estery Compounds	Citrus-Piney Compounds
(Caryolan-1-ol)[a]	Geraniol	Δ-Cadinene
Caryophyllene Oxide	Geranyl Acetate	γ-Cadinene
(Humulene Diepoxide A)	Geranyl Isobutyrate	(Citral)
Humulene Diepoxide B	Linalool	Limonene
(Humulene Diepoxide C)		(Limonene-10-ol)
Humulene Epoxide I		α-Muurolene
Humulene Epoxide II		(Nerol)
Humulene Epoxide III		β-Selinene
Humulenol II		
Humulol		

[a]Compounds not usually detected in steam-distilled oil from fresh hops are in parentheses.

The OSU samples were machine-picked, dried to 8–10% moisture, and compressed into miniature bales (20 × 10 × 15 cm, 600 g). The bales were put into plastic bags and stored at −40°C until analysis. Miniature bales were halved, and 300-g samples were placed in ambient storage (20–25°C) for six months before analysis. Sealed commercial pellet samples (20 kg) were opened and analyzed within two to three days.

A modified oil trap (Fig. 1) adapted from Burkhardt (4) was used to collect and measure the hop oil. The modified oil trap minimizes the contact between hot steam and condensed oil by changing the position of the steam input and by using a cold-finger condenser. Dried hop cones or pellets (250 g) were steam distilled for 5 hr from 5 L of buffered tap water. A phosphate buffer (0.05*M*, pH 6) of 34 g of KH_2PO_4 and 50 ml of 0.67*N* NaOH in 5 L of water was used. For green hops, a sample weight of 600 g was used. An internal standard, 0.025 ml of tetradecane, was added to all samples before distillation. Immediately after distillation, the volume of oil was measured, transferred to glass ampoules, sealed, and stored at −4°C. For GC, a 0.1-ml aliquot was transferred to a 1.8-ml autosampler vial, and 1.0 ml of pentane was added.

Wort

Cooled wort samples were collected in 3.78-L (1-gal) brown glass jugs with foil-lined caps. Five milliliters of 40% formaldehyde was added to each container as a preservative. Formaldehyde was selected because it is not detected by flame ionization detectors. The wort samples were stored at 4°C until analysis. The Likens-Nickerson steam-distillation/extraction apparatus (16) was used to collect the volatiles from 1.0 L of wort. Pentane, 25 ml, was used as the extracting solvent. Bumping was a problem with high-gravity worts. Several surfactants were tried, and most resulted in the production of long-chain volatile fatty acid esters. Consequently, to eliminate artefact production, no surfactants were used to control bumping. Instead, the wort was diluted 1:1 with distilled water. Potassium acid phosphate (KH_2PO_4, 0.05*M*, 3.6 g) was added to each 2-L sample and adjusted to pH 6 with 1.0*N* sodium hydroxide. The internal standard, tetradecane (0.0005 ml), was added with a 0.5-μl syringe. After slowly heating to boiling, the sample was distilled and extracted for 90 min. Boiling at high temperature for more than 120 min resulted in compounds that were steam-distillable but had very long retention times (several hours) with our gas chromatographic conditions.

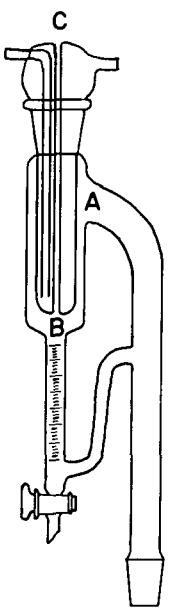

Fig. 1. Modified trap for steam distillation of hop oils. The steam vapor (A) does not heat the condensed oil (B), and the vent (C) prevents bumping.

The pentane from the trap and distillation flask was combined, frozen to remove water (−10°C), and concentrated to 0.1–0.2 ml with nitrogen at less than 30°C in a water bath. The extract was transferred to an 0.1-ml autosampler vial with a Teflon-lined septum and stored at −4°C until GC analysis.

Beer

Six bottles or cans (2.13 L) were used for each extraction. The exterior of the crowns were washed with acetone before uncapping. The crowns then were placed in the flask with the beer so that any oil constituents absorbed by the cap liner would be extracted (22). The solutions were adjusted to pH 6 with 1.0*N* NaOH after 14.5 g of KH_2PO_4 was added. They then were distilled for 90 min. The collection tube was filled with 70% ethanol in distilled water. Water was added to the pentane extract so that the ethanol and pentane phases separated and most of the aqueous ethanol was removed by pipet before chilling. The pentane extract was concentrated in the same manner as was the wort concentrate.

Hop Oil Fractionation

Hop oil hydrocarbon and oxygenated fractions were separated on a 100- to 200-mesh silica gel column containing 14% water. A 0.1-ml aliquot was placed on a 3-mm × 8-cm dry column and hydrocarbons were eluted with 10 ml of pentane. Oxygenated compounds then were eluted with 10 ml of anhydrous diethyl ether. The hydrocarbon fraction already contained the internal standard, so 0.5 μl of tetradecane was added to the oxygenated fraction after separation. Both solutions were concentrated to about 1 ml for GC analysis.

Gas Chromatographic Conditions

An HP 5890A (Hewlett-Packard, Palo Alto, CA) gas chromatograph with a flame ionization detector, HP 7672A autoinjector, and HP 3393 integrator was used. The column was a 60-m (0.25-mm-i.d.) Supelcowax-10 bonded 0.25-μm film fused silica capillary column (Supelco #2-4081). The injection port temperature was 230°C, and the detector temperature was 250°C. Helium was the carrier gas with a flow rate of 0.7 ml/min and split ratio of 1:50. Nitrogen was used as a makeup gas (15 ml/min). The autoinjector was set to inject 2 μl for hop oils and 5 μl for worts and beers.

Temperature Program

The column temperature was programmed at 80°C for 5 min, 80–160°C at 5°C/min, 160–240°C at 4°C/min, and held at 250°C for 24 min (hop oils) or for 59 min (wort and beer). Since 1987, we have used several columns and found that only minor changes in the temperature program were needed to produce similar

TABLE II
Precision of Hop Aroma Profile Measurement[a]

	Hop Aroma Component Concentration, nl/g or μl/L			
Sample	Oxidation Products	Floral-Estery	Citrus-Piney	Aroma Units
GLC				
Mean	47.47	108.51	246.48	402.45
SD	± 6.449	± 0.049	± 1.845	± 8.245
Hops				
Mean	555.84	152.84	649.46	1,358.14
SD	± 65.856	± 24.019	± 30.702	± 120.577
Wort				
Mean	0.0782	0.1043	0.0029	0.1854
SD	± 0.0114	± 0.0134	± 0.0001	± 0.0247
Beer				
Mean	0.0140	0.0312	0.0028	0.0473
SD	± 0.0048	± 0.0041	± 0.0011	± 0.0099

[a]Mean and standard deviation (SD) of duplicate hop oil injections (GLC), steam-distilled hop oil from two commercial bale plugs (hops), and duplicate steam/distillation/extraction of volatiles from wort and beer.

retention times. For example, changing the second temperature increase from 3.5 to 4°C per minute gave the same retention times for a new column as did a year-old column. The total analysis time for hops was 65 min. Wort and beer samples required 100 min to elute nonhop volatiles. GC-mass spectrometry and purified compounds were used to verify peak identification.

Calculations

Relative retention times (retention time of the compound divided by the retention time of the internal standard) were calculated for each peak. The raw area percent data were corrected for the added amount of internal standard. The hops amounts (nanoliters per gram) were calculated from the measured oil content and the corrected GC area percent in the oil. The relative response for each compound was not used in the calculations. The amount of each compound in wort and beer was calculated from the internal standard. The amount reported for wort analysis was adjusted for the final dilution of the beer.

RESULTS AND DISCUSSION

Although the sensitivity and selectivity of the methods used for analyzing hops has increased enormously in the last 20 years, obtaining a representative sample is still the major problem when whole-hop cones are analyzed. For certified hop analysis of baled hops, many core samples are composited, ground, and subsampled for the determination of α- and β-acids and moisture content (1,2). Unfortunately, the hop oil content of ground hops decreases dramatically, even during frozen storage (3). In our laboratory, we determine the oil content by steam distillation of more than 1,000 whole-cone samples each season and have found it impractical to grind each sample. Other methods, such as solvent extraction (15,29) and headspace analysis (18), have advantages over steam distillation in that there is no thermal degradation or possible hydrolysis of terpene esters (25). These methods also have disadvantages—solvent extracts have to be concentrated, nonvolatiles have to be removed, and headspace analysis does not usually detect compounds with boiling points higher than the boiling point of humulene (18). This includes most of the sesquiterpene oxidation products and esters, which have higher boiling points and longer GC retention times than humulene. One of the disadvantages of steam distillation is the time required for distillation (15). This is misleading. The operator time required for steam distillation actually is shorter than the time required for solvent extraction. Steam distillation produces enough hop oil for further separation and isolation of individual components or fractions.

The compounds included in the HACP were selected on the basis of reported contribution to hop and beer aroma. These compounds usually are present in hop oil at sufficiently high concentrations and can be accurately measured by gas chromatographic analysis. The data in Table II show the results of duplicate injections of each of two steam-distilled hop oils from brewer's inspection samples, i.e., plugs cut from commercial bales. Replicate GC injections of a single hop oil had an average 2% coefficient of variation, whereas the bale plugs had a much higher 8.9% variation. Pellet samples actually are composited before pelletizing, and results are less variable (3). The samples from the OSU world hop collection were composited from five to 10 plants during picking, and there was less variation between miniature bales than there was between commercial bales. The results for duplicate analyses of worts and beer also are shown in Table II. Although the wort and beer samples were more homogeneous than hops, the results show greater variation between duplicate analyses. Even though detection sensitivity has increased several thousand times in the last 25 years, the quantitation of amounts of volatile compounds (ppb) is still difficult.

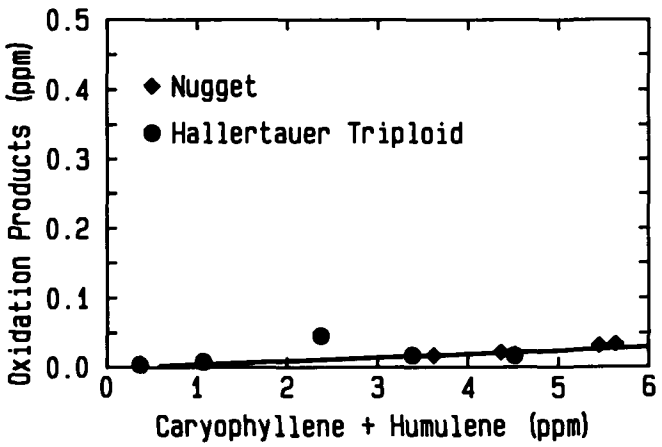

Fig. 2. Formation of caryophyllene and humulene oxidation products during steam distillation of green hops.

Humulene and caryophyllene oxidation products increase during storage (5,14). Drying and baling also may affect the amounts of oxidation products, and oxidation may occur during steam distillation. When dried hops are analyzed, it is not possible to determine whether or not oxidation products are the result of steam distillation or storage. The effects of drying and baling can be studied by analyzing green hops. The amount of humulene and caryophyllene oxidation products increases with the amount of humulene and caryophyllene present in green hops as shown in Figure 2, but the oxidation products are only 8% of the original amount of humulene and caryophyllene and less than 0.2% (50 nl/g) of the total oil content.

Table III shows the results for two varieties analyzed periodically before harvest. The HACP increased during maturity but leveled off once the highest oil content was reached. Although the oil and α-acids content of these varieties were quite different, the HACP totals (AU) were similar and the relative proportions

TABLE III
Effect of Maturity on Hop Aroma Component Profile Composition

Variety[a]	Picking Date	Hop Aroma Components Concentration, nl/g: Oxidation Products	Floral-Estery	Citrus-Piney	Aroma Units
USDA 21459	August 10	21.8	3.3	63.8	88.9
	August 16	74.5	12.6	273.1	360.2
	August 30	49.4	93.9	615.1	758.4
	September 7	162.5	162.6	908.4	1,233.4
	September 13	123.3	240.2	912.6	1,276.1
Nugget	August 15	217.3	116.2	539.6	873.1
	August 22	297.3	195.1	648.1	1,140.5
	August 29	272.3	189.0	583.9	1,045.2
	September 5	127.9	241.2	525.4	894.5

[a]USDA 21459 is a triploid Hallertauer variety and is naturally seedless. Nugget is a high α-acid variety and is seeded in Oregon.

TABLE IV
Hop Aroma Component Profile[a] of Mt. Hood from Commercial and Experimental Plots

Hop Aroma Components	Concentration, nl/g: Commercial Oregon	Experimental Plots: Fresh	Aged
Oxidation products	114.0	119.5	1,245.4
Floral-estery	66.2	63.2	69.6
Citrus-piney	321.7	473.7	441.8
Aroma units	501.9	656.4	1,756.8

[a]Mean of duplicate analyses. Samples from 1988 crop year. The aged samples were stored at ambient temperature for six months.

of floral-estery and citrus-piney compounds were consistent for each variety during maturity. A comparison of the HACPs of Mt. Hood from a commercial yard and an OSU experimental plot (Table IV) showed a 20% difference in the total AU content of the fresh samples due to a difference in the citrus-piney compounds. The increase of AU of the experiment plot with aging is attributable to the increase in oxidation products.

Table V shows the changes in HACP that occur as a yard matures. Mt. Hood was planted in 1986 and first harvested in 1987. The 1989 analyses showed much greater agreement between the HACPs for each state than did the first harvest sample.

Samples from the hop variety collection show considerable year-to-year variation in the amounts of hop oil collected (Table VI). For example, the amount of oil found in Cacades ranged from 0.28 to 1.79 ml of oil per 100 g of hops, with an average value of 1.27 (1975–1985 analyses). The percent myrcene in the oil varied from 46 to 82% of the oil or the equivalent of 3,276 to 11,817 nl of myrcene per gram of hops. However, the ratio of humulene to caryophyllene only varied from 2.58 to 3.07, and the ratio of humulene to farnesene varied from 1.89 to 2.07. Except for the 1982 Cascade results, AU values increased with age. Generally, it is the increase of oxidation products with storage that increases AUs. However, some oil components decrease during aging so that the perceived aroma of aged hops may be different than that of a fresh sample. Also, there is a varietal difference in the keeping qualities of hops. For example, in 1989, Brewers Gold lost more α-acids during our six-month storage trial than did Nugget. The total AUs for the fresh samples were similar, but the Brewers Gold aged AUs were twice those of Nugget. However, if the amount of oil components are divided by the calculated original α-acids content (Sigma), then aged Brewers Gold has three times the amount of volatiles per gram of α-acid than aged Nugget does.

Pellet hops are preferred by a number of breweries because of their convenient packaging and reduced storage losses. Table VII shows the oil composition of several varieties of pelleted hops. Pellet samples from the same crop year came from different suppliers. The total AUs found in Cascade and Hallertauer pellet samples were close to the values found for the aged OSU samples. Two factors may have contributed to the increase in oxidation products and total AUs. First, the samples may have been stored for several months before processing, and second, the process of grinding and pelletizing may have oxidized the oil.

TABLE V
Hop Aroma Component Profiles of Commercial Mt. Hood Samples Grown in 0.8-ha Plots in Three States[a]

Location	Crop Year	Oxidation Products	Floral-Estery	Citrus-Piney	Aroma Units
Idaho	1987	186.2	168.9	400.8	755.9
	1988	574.9	189.1	473.9	1,237.9
	1989	550.2	178.0	558.4	1,286.6
Oregon	1987	207.6	110.8	433.5	751.9
	1988	114.0	66.2	321.7	501.9
	1989	555.8	152.8	649.5	1,358.1
Washington	1987	116.4	19.4	128.3	264.1
	1988	87.8	51.3	265.2	404.3
	1989	433.0	153.0	426.4	1,012.4

[a]The yards were planted in 1986 and the 1987 results are from the first or "baby" crop and are different from a mature crop.

CONCLUSIONS

Comparative methods for determination of volatiles in hops, wort, and beer have been described. The results of analyzing several varieties of hops for their HACP show that there is considerable variation in the quantitative amounts, although the ratios of selected compounds remain varietal characteristics. Hop oil analyses that emphasize varietal identification do not provide results that the brewer can use to control hop aroma because the major hop oil constituents are not present in beer. The HACP and the AU concept provide a measurement with direct application. Just as the actual α-acid content of a particular variety

TABLE VI
Hop Aroma Component Profiles of Varieties from OSU[a] Variety Collection

		Concentration, nl/g							
		Fresh				Aged (six months ambient temperature)			
Variety	Year	Oxidation Product	Floral-Estery	Citrus-Piney	Total AU[b]	Oxidation Product	Floral-Estery	Citrus-Piney	Total AU
Brewers Gold	1982	150	116	653	920	488	77	298	863
	1989	154	550	327	1,031	1,422	509	334	2,273
Cascade	1982	462	194	341	997	185	52	70	307
	1989	62	75	252	389	528	105	250	883
Fuggle	1982	204	83	286	573	532	63	168	764
	1988	55	32	149	236	105	23	165	293
	1989	89	57	228	374	219	54	245	518
Hallertauer	1982	85	65	233	383	408	70	190	668
	1989	83	96	258	437	619	75	228	922
Nugget	1982	158	113	577	848	569	107	384	1,060
	1987	232	101	462	795	288	96	390	774
	1989	159	261	644	1,064	449	248	568	1,265
Olympic	1982	135	40	322	497	304	73	145	522
	1989	28	261	174	463	361	294	182	837
Perle	1982	123	83	408	614	384	85	276	745
	1989	43	80	191	314	749	164	455	1,368
Styrian	1982	149	48	201	398	434	56	104	594
	1988	32	46	88	166	102	58	141	301
	1989	74	115	252	441	598	103	305	1,003
Willamette	1982	111	53	374	538	431	58	206	695
	1986	110	122	285	517	244	100	210	554
	1989	71	83	298	453	414	124	284	922

[a]Oregon State University.
[b]Aroma units.

TABLE VII
Hop Aroma Component Profile of Hop Commercial Hop Pellets[a]

		Hop Aroma Component Concentration, nl/g			
Variety	Crop Year	Oxidation Products	Floral-Estery	Citrus-Piney	Aroma Units
Cascade	1986	384.5	302.0	288.2	974.7
	1986	136.2	592.3	311.9	1,040.4
	1987	276.9	309.0	474.6	1,050.4
	1987	169.8	197.5	385.8	953.1
	1987	227.1	46.4	356.7	630.2
	1988	233.9	180.1	334.0	748.0
	1988	152.8	134.5	426.4	713.7
Chinook	1986	220.7	607.0	1,115.6	1,943.3
	1988	277.2	202.1	1,391.6	1,870.4
Cluster	1986	37.0	29.0	1,391.2	453.6
	1986	75.4	138.8	387.6	488.7
	1987	90.2	66.8	274.5	328.2
	1987	112.2	98.1	171.2	427.8
	1987	151.4	45.8	217.5	438.3
	1987	148.7	19.8	241.1	401.0
	1989	90.1	119.0	232.5	430.0
Hallertauer	1988	313.0	98.6	220.9	882.3
	1989	242.9	150.2	470.7	869.6
Hüller Bitter	1985	152.7	72.3	476.5	925.8
	1985	175.0	87.5	700.9	928.7
	1987	519.2	34.3	666.2	1,062.5
Saazer	1987	243.8	28.1	509.0	380.8
Tettnanger	1987	258.6	29.0	108.9	614.0
	1987	473.4	49.8	326.4	764.7
	1987	942.9	59.9	241.5	1,393.6
	1988	420.0	43.9	390.8	623.3
	1989	135.0	163.7	159.4	603.3
	1989	203.8	33.2	394.6	399.6

[a]Each analysis is from a different lot.

is used to adjust hopping rates, the AU content may be used to adjust a beer's hop aroma.

The relative proportions of oxidation products, floral-estery compounds, and citrus-piney compounds may play some part in the hand evaluation of certain hop varieties as acceptable aroma hops. A measurement of potential hop aroma provides the brewer an objective rather than a subjective test for evaluating hops. The HACP also can be used to monitor quality during processing. The effects of maturity, drying, baling, and pelletizing can be controlled so that a more consistent product is obtained.

For 25 years, scientists have tried to identify the compound responsible for hoppy character in beer without success. Hoppy aroma in beer is probably not attributable to a single component but rather to the synergistic effect of several compounds. Minor constituents produced by fermentation of hop oil constituents may be a part of this hoppy character. The actual hop oil constituents that compose the HACP may change as future research reveals new compounds that contribute to the hoppy character in beer. But the principle remains—measure the constituents present in hops and use this information to adjust the hopping rates for consistent hoppy character.

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