



Influence of variety, wine style, vintage and viticultural area on selected chemical parameters of Canadian Icewine

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Received 5 May 2007, accepted 8 August 2007.

Abstract

Icewine is a premium Canadian product made from grapes that have been allowed to freeze naturally on the vine. When pressed in their frozen state, they yield juice and finally wine highly concentrated in sugar and flavour compounds. This study surveyed a comprehensive range of Icewines (348) and reports on their chemical composition and how this is mediated by variety (Vidal, Riesling, Gewurztraminer, Chardonnay, Cabernet Franc and Cabernet Sauvignon), vintage (2000-2004), province-of-origin (Ontario, British Columbia and Nova Scotia) and style ('standard', sparkling and oaked). In addition, data on icewine juice °Brix (JB) was collected where possible, and related to wine composition. Wines from British Columbia had the highest residual sugar (RS) and those from Nova Scotia the lowest, while free SO₂, total SO₂ and sorbic acid also differed between provinces. Vintage was a source of variation for JB, RS and sorbic acid (SA). Grape variety influenced JB and volatile acidity (VA), and VA and SA varied with wine style. A number of analytes were positively correlated, including JB, VA and RS. We conclude there is stylistic divergence between the provinces and that Icewine is a complex and heterogeneous wine style.

Key words: Ice wine, icewine, Eiswein, chemical composition, Riesling, Vidal, Ontario, British Columbia, Nova Scotia.

Introduction

Ice wine is produced from grapes that have been frozen on the vine and pressed while still frozen, resulting in a dessert wine highly concentrated in sugar and flavour compounds. Germany and Austria have traditionally been the main producers (where it is known as Eiswein), however, Canada is now the world's dominant player, with her total ice wine production now in the order of 1,000,000 L¹. The term 'Icewine' is trademarked and refers to ice wine that has been produced in Canada according to strict standards of quality control, enforced by the Vintners Quality Alliance (VQA), the enactment of which came into force in 2000. Quality parameters such as temperature at harvest ($\leq -8^{\circ}\text{C}$), juice °Brix (average ≥ 35) and minimum wine residual sugar (125 g L⁻¹) are regulated under VQA, and these standards have contributed significantly to the success of the style¹. However, to date, only Ontario (ON) and British Columbia (BC) subscribe to the VQA program; thus ice wines produced in other provinces need have no minimum quality regulations.

Canada's viticultural regions are spread across a continent – from the Pacific province of BC, to Nova Scotia (NS), on the Atlantic. However, it is in ON (approx 75%) and BC (approx 24%) where most Icewine production occurs. Here, often in contrast with many European ice wine producing countries, sufficiently cold temperatures occur at the correct time and for long enough to harvest and press the frozen fruit. The predominant grape varieties are Vidal and Riesling, which possess the requisite physiological properties to withstand the rigours of the climate,

and together account for approximately 90% of the Icewine harvest. However, in recent years other provinces have added Icewine to their portfolios and new varieties and styles have emerged, yet to be characterized in the literature.

Cliff *et al.*² determined the sensory and compositional profiles of 25 Canadian and German ice wines using descriptive, physicochemical and volatile analyses. They found eight volatile compounds differed significantly between BC and ON Icewines and noted differences between regions or countries for viscosity, titratable acidity, total sugar, colour absorbance values and ethanol. German, ON and BC wines also showed sensory differences for aroma, taste, body/viscosity and colour attributes. Nurgel *et al.*³ evaluated 51 Icewines chemically and twenty sensorially, and reported that wines from ON and BC were significantly different for a range of chemical and sensory attributes. They also found differences between Riesling and Vidal and between vintages for a number of physico-chemical properties. However, the authors acknowledged limitations in the study from the relatively small sample size and incomplete regional representation and encouraged further research to address this.

To our knowledge, these are the only two surveys of commercial Icewine composition that appear in the literature, and neither reports on Icewine juice composition. The main objectives of our study therefore were to (i) characterize the composition of a comprehensive range of Icewines, including examples of the more recent varieties and styles, and (ii) investigate the possible

mediating influence of province-of-origin and vintage. A secondary objective was to collect and describe summary data on Icewine juice Brix, where available, and relate this to wine composition.

Materials and Methods

Wines were obtained by the Liquor Control Board of Ontario (LCBO) and analyzed as part of their quality control program. All samples were analyzed simultaneously for ethanol, free SO₂ (FSO₂), total SO₂ (TSO₂), SA, RS and VA using a multi-channel Skalar San Plus segmented flow analyser equipped with an SA1074 autosampler, an SA5501 circulating water bath and SA5521 reactors. All channels were interfaced to individual SA6250 matrix photometers and data were collected by an IBM PC using SAN^{plus}SYSTEM software Flow Access^R, Version 1.04.4, all manufactured by Skalar Systems, Breda, Netherlands. Each component was analyzed based on the following principles:

Sorbic acid: Samples were distilled into an alkaline solution. Potassium dichromate was added to oxidize the SA to malondialdehyde, forming a red coloured complex with thiobarbituric acid which was measured at 520 nm.

Volatile acid: Determination was based on a pH shift; the samples were mixed with tartaric acid and distilled at 96°C with nitrogen gas as a carrier flow. Hydrogen peroxide was added and the samples were re-sampled in a buffered indicator solution (phenolphthalein). The distilled acids cause a pH shift and decolourisation of the phenolphthalein colour reagent which was measured at 550 nm.

Total residual sugar: Samples were hydrolysed at 95°C, mixed and dialysed against a sodium carbonate solution. After addition of a copper neocuproin mixture, the stream was heated to 97°C. The copper neocuproin chelate is reduced by the presence of sugars, forming a yellow cupro-neocuproin complex that was measured at 460 nm.

Free sulphur dioxide: Samples were acidified with hydrochloric acid. Gaseous sulphur dioxide was formed and dialysed into a sulphuric acid solution. A formaldehyde solution and a pararosaniline solution were added. A red coloured complex is formed by the reaction of the sulphur dioxide, formaldehyde and the pararosaniline. The absorption was measured at 560 nm.

Total sulphur dioxide: Samples were diluted in a sulphuric acid solution and heated to 95°C to release the bound sulphur dioxide. Gaseous sulphur dioxide is formed which was dialysed into a sulphuric acid solution. The analysis proceeded exactly as in the procedure described for FSO₂.

Ethanol: Determination of ethanol is based on the chromium reduction reaction. The sample was distilled at 95°C, re-sampled and mixed with a potassium chromate solution. Cr (VI) is reduced in the presence of ethanol to Cr (III). The green coloured Cr (III) complex was measured at 590 nm.

JB was measured for ONT samples only by individual wineries as part of the recording procedures required and enforced by the Vintners Quality Alliance of Ontario (VQAO).

Data for all analytes was analyzed using the ANOVA procedure within XLSTAT[®] version 7.5.2 (Addinsoft, 40, rue Damrémont, 75018 Paris, France).

Results

Overall: The grand means for each juice and wine quality parameter are given at the bottom of Tables 1 and 2. The range of values and

median scores were also calculated for each analyte and are, respectively: JB 35.0–46.1, 38.8; ethanol (% v/v) 6.5–14.4, 10.7; RS (g L⁻¹) 70–307, 208; VA (g L⁻¹) 0.11–2.35, 1.14; FSO₂ (mg L⁻¹) 0.0–72, 20; TSO₂ (mg L⁻¹) 14.0–510, 176; SA (mg L⁻¹) 0.0–279.0, 0.0. Pearson's correlation coefficient and corresponding probability were calculated for each pair of analytes and are shown in Table 3.

Sources of variation: Table 1 shows the influence of grape variety and vintage on juice and wine composition of the samples. ANOVA was also performed on the data with province and vintage as independent variables. If the F-statistic was significant ($p < 0.05$), Fisher's LSD was then applied as the means separation test. As shown, province was a significant source of variation for four analytes: RS ($F = 6.27$, $p = 0.002$), FSO₂ ($F = 6.75$, $p < 0.001$), TSO₂ ($F = 6.37$, $p < 0.002$) and SA ($F = 4.60$, $p = 0.011$). Vintage was a significant source of variation for three analytes: JB ($F = 4.18$, $p = 0.003$), RS ($F = 3.36$, $p = 0.010$) and SA ($F = 5.95$, $p = 0.000$).

Table 2 shows the variation in concentrations of the various quality parameters for the different varieties and wine styles. ANOVA was also performed on the data with variety and style as independent variables. The varietal analysis excluded the 'other whites' and 'other reds' data. If the F-statistic was significant ($p < 0.05$), Fisher's LSD was then applied as the means separation test. As shown, variety was a significant source of variation for JB ($F = 3.41$, $p = 0.005$) and VA ($F = 7.52$, $p < 0.0001$), while ethanol approached significance ($F = 2.23$, $p = 0.051$). Data for the 'other white' and 'other red' wines were re-examined, given their low n and therefore the potential for atypical samples to have a greater effect on the group mean. One Merlot had an exceptional high RS concentration (299 g L⁻¹); it was removed and the group average recalculated as 224.8±6.0 g L⁻¹. Additionally, one Merlot juice was measured at 42.1 Brix; after its removal, the average JB for 'other reds' was recalculated as 39.37±1.99.

Icewine style was a significant source of variation for VA ($F = 7.46$, $p = 0.001$) and SA ($F = 11.70$, $p < 0.0001$), although ethanol ($F = 2.51$, $p = 0.083$) and JB ($F = 2.63$, $p = 0.074$) approached significance. To further investigate the influence of style, comparisons were made between white and red Icewines using ANOVA (excluding sparkling and oaked wines). The only analyte that differed was ethanol ($F = 4.28$, $p = 0.040$), where red wines (mean = 10.92% v/v) had a higher concentration than white wines (mean = 10.57% v/v).

Discussion

General observations: As shown in Tables 1 and 2, Icewine juices are characterized by much higher sugar concentration (average JB = 39) than that used for table wines or most other dessert wine styles, and the wines have much higher RS (average of 207 g L⁻¹). This concentration of sugars occurs from the freezing of most of the water content of the berry on the vine, and its retention in the press as ice crystals during pressing operations¹. Juice soluble solids concentration is largely dependent on the temperature at pressing, which typically is in the -9 to -11°C range in Ontario and can be predicted using the formula of Wuerdig *et al.*⁴.

The higher VA and lower ethanol concentration compared to table wines is likely due to the challenges experienced by the fermenting yeast in the Icewine juice matrix. Specifically, the high soluble solids concentration creates hyperosmotic stress on the yeast, which limits growth and the degree of ethanol conversion

Table 1. Provincial and vintage variation in quality indicators for Icewine.

	Must °Brix ¹	Residual sugar (g L ⁻¹)	Ethanol (% v/v)	Volatile acidity (g L ⁻¹)	Free SO ₂ (mg L ⁻¹)	Total SO ₂ (mg L ⁻¹)	Sorbic acid (mg L ⁻¹)
Province							
Ontario (n=335)	39.0± 2.1	207 b± 36	10.7 a± 1.3	1.18 a± 0.28	22 b± 16	179 a± 72	40.58 a± 70.38
British Columbia (n=8)	ND	233 c± 44	10.0 a± 1.6	1.12 a ±0.23	7 a ± 7	133 a± 34	97.63 b± 81.70
Nova Scotia (n=5)	ND	159 a± 60	10.0 a± 0.7	1.01 a± 0.07	37 c± 14	306 b± 72	136.67 b± 4.73
Vintage							
2000 (n=26)	39.8 b± 2.4	225 b± 40	10.2 a± 1.3	1.14 a± 0.25	20 a± 18	172 a± 50	102.05 b± 79.55
2001 (n=76)	38.1 a± 2.0	204 a± 46	10.5 a± 1.4	1.15 a± 0.28	20 a± 16	173 a± 66	42.06 a± 74.60
2002 (n=96)	39.1 b ±2.1	215 b± 32	10.5 a± 1.3	1.19 a± 0.26	23 a± 94	163 a± 53	27.56 a± 59.69
2003 (n=59)	39.1 b± 2.1	199 a± 25	10.9 a± 1.4	1.16 a± 0.28	18 a± 14	176 a± 76	33.73 a± 63.02
2004 (n=91)	39.3 b ± 2.0	200 a± 38	10.9 a± 1.1	1.23 a± 0.29	24 a± 15	207 a± 93	49.15 a± 74.70
Overall (n=348)	39.0 ± 2.1	207 ± 37	10.6± 1.3	1.18± 0.27	21± 16	179± 73	42.83± 71.33

¹ data available for Ontario only; data represent means ± standard deviations; for each analyte, means with the same letter do not differ significantly between province (a,b,c) or vintage (a,b,c) (Fisher's Protected LSD _{0.05}).

Table 2. Influence of grape variety and wine style on Icewine quality indicators.

	Must °Brix ¹	Residual sugar (g L ⁻¹)	Ethanol (% v/v)	Volatile acidity (g L ⁻¹)	Free SO ₂ (mg L ⁻¹)	Total SO ₂ (mg L ⁻¹)	Sorbic acid (mg L ⁻¹)
Variety							
Vidal (n=171)	38.8 a± 2.0	203 a± 35	10.7 a± 1.3	1.10a± 0.22	22 a± 16	179 a± 66	48.01 a± 72.96
Riesling (n=69)	38.8 a± 2.2	210 a± 42	10.2 a± 1.3	1.23 b± .31	18 a± 12	172 a± 72	38.98 a± 69.91
Gewurztraminer (n=20)	40.4 b± 2.6	207 a± 36	10.7 a± 1.0	1.40 c±0.31	27 a± 21	190 a± 73	41.55 a± 72.33
Chardonnay (n=7)	41.2 b± 3.5	236 a± 23	11.0 a± 1.1	1.34 bc±0.36	19 a± 13	230 a± 114	52.67 a± 81.89
Other whites ² (n=7)	40.7± 1.8	215± 47	9.9± 0.9	1.38± 0.36	25± 22	284± 168	55.40± 75.90
Cabernet Franc (n=57)	38.6 a± 1.9	206 a± 39	10.9 a± 1.3	1.19 b± 0.24	21 a± 15	163 a± 57	34.18 a± 70.94
Cab. Sauvignon (n=12)	38.9 a± 1.7	207 a± 17	10.8 a± 1.4	1.24 bc± 0.33	26 a± 18.0	186 a± 93	10.27 a± 34.07
Other reds ³ (n=5)	40.1± 2.1	240± 33	10.8± 1.2	1.35± 0.35	26± 14	198± 98	64.60± 88.46
Style							
Standard ⁴ (n=330)	38.9 a± 2.1	206 a± 37	10.7 a± 1.3	1.18 b± 0.27	22 a± 16	180 a± 73	41.78 a± 70.59
Sparkling ⁴ (n=9)	ND	205 a± 29	9.7 a± 0.9	0.90 a± 0.17	16 a± 5	203 a± 58	156.33 b±19.25
Oaked ⁵ (n=9)	40.5 a± 1.0	231 a± 23	10.8 a± 1.0	1.37 c± 0.20	19 a± 15	164 a± 85	70.44 a± 84.04
Overall (n=348)	39.0 ± 2.1	207 ± 37	10.6± 1.3	1.18± 0.27	21± 16	179± 73	42.83± 71.33

² 2 Chenin blanc, 1 Geisenheim, 1 Muscat Ottonel, 1 Muscat, 1 Ortega, 1 Viognier; ³ 3 Merlot, 1 Pinot noir, 1 Shiraz; ⁴ 7 Vidal and 2 Riesling; ⁵ all Vidal; ⁶ non-sparkling and un-oaked wines; data represent means ± standard deviations; for each analyte, means with the same letter do not differ significantly between variety (a,b,c) or style (a,b,c) (Fisher's Protected LSD _{0.05}).

and initiates the hyperosmotic glycerol (HOG) response, resulting in the production of elevated levels of acetic acid ⁵, the major constituent of VA. However, the average value reported here for VA is below the legislated limit in Canada (2.4 g L⁻¹) and the reported sensory threshold in Icewine of 3.2 g L⁻¹ ⁶. Our average RS concentration (206.7 g L⁻¹) falls between that previous reported by Cliff *et al.*² (202.2 g L⁻¹) and Nurgel *et al.*³ (214.7 g L⁻¹), while our average ethanol concentration (10.6% v/v) is higher than that found by Cliff *et al.*² (10.1% v/v) and Nurgel *et al.*³ (10.2% v/v). The VA data reported here (average 1.2 g L⁻¹) is in good agreement with the average acetic acid concentration found by Nurgel *et al.*³ (1.3 g L⁻¹).

The SA values reported here likely under represent the 'true' average concentration for Icewine; many of the samples were analysed some days prior to bottling, whereas sorbate is often adjusted or added for the first time at or immediately prior to bottling (as an antimicrobial agent) ⁷. Thus, we recalculated the SA data after removing all zero values, and obtained a mean concentration of 140.4±54.4 mg L⁻¹, and a median of 158.0 mg L⁻¹. This is in good agreement with the mean concentration for wines that were analysed as part of the LCBO export certification process, and thus assumed to be in a finished state (154.6±11.2 mg L⁻¹). Similarly, but to a lesser extent, caution is advised in interpreting the SO₂ values, as these are also often adjusted at bottling. However, in general the SO₂ concentrations appear similar to those reported for table wines.

Very little research appears in the English literature allowing for

comparisons with the composition of ice wines from other countries. Cliff *et al.*² reported RS and ethanol concentrations of 166.3 g L⁻¹ and 9.0% v/v, respectively, for German Eiswein; significantly lower than reported here for Icewine. However, with a sample size of only three, strong caution is advised in over interpreting their data, although it is consistent with much anecdote suggesting that German and Austrian Eiswein are produced from juice of lower sugar concentration than used for Icewine.

Correlations: JB is positively correlated with RS and VA (Table 3). A positive association has previously been reported between Icewine JB and wine VA concentration in lab trials ⁵, perhaps suggesting that as the hyperosmotic stress on the yeast increases with increasing sugar concentration, the HOG response produces proportionally more acetic acid. However, Inglis *et al.*⁸ report that while the amount of acetic acid produced as a proportion of sugar consumed increases linearly across the range of 40–46 Brix, yeast in higher Brix juices ferment less sugar, resulting in approximately the same VA concentration in the final wines. As expected, RS is inversely correlated with ethanol concentration. This result is likely due to the inhibitory effect on alcoholic fermentation of high Brix juices, given the negative association between JB and wine ethanol concentration (r = -0.112; p = 0.054). The positive correlation between RS and VA may also be reflecting alterations in yeast metabolite production as juice Brix increases. As expected, FSO₂ is strongly and positively associated with TSO₂ and, less strongly with SA concentration.

Table 3. Pearson correlation matrix for juice and Icewine quality parameters.

	Juice Brix	Residual sugar	Ethanol	Volatile acidity	Free SO ₂	Total SO ₂	Sorbic acid
Juice Brix	1	0.384	-0.112	0.406	0.001	0.014	-0.077
RS	0.384	1	-0.472	0.297	0.040	-0.031	-0.015
Ethanol	-0.112*	-0.472	1	0.031	-0.025	-0.103	-0.052
VA	0.406	0.297	0.031	1	-0.077	-0.076	-0.033
Free SO ₂	0.001	0.040	-0.025	-0.077	1	0.739	0.126
Total SO ₂	0.014	-0.031	-0.103	-0.076	0.739	1	0.107
Sorbic acid	-0.077	-0.015	-0.052	-0.033	0.126	0.107	1

Data based on 298 samples; coefficients in bold are statistically significant at $p < 0.0001$ except for 0.126 ($p = 0.030$); * $p = .054$.

Provincial and vintage variation: NS wines had on average 28% lower RS than those from ON and BC while retaining similar ethanol concentration, which suggests they were made from lower JB. While the temperature at pressing is the strongest determinant of the final sugar concentration of Icewine juice⁷ the shorter growing season in NS and/or the presence of some atypical Icewine varieties in this sample (17% Geisenheim, 17% Ortega, 17% Muscat) may be influencing this result. These data may inform the discussion on whether adoption of VQA quality regulations outside of ON and BC should be pursued for Icewine. The higher RS for BC wines compared to those from ON is likely not explained by the different varietal mixes. While the ON samples are predominantly Vidal, and those from BC are predominantly Riesling (reflecting actual distribution⁷), these varieties tend to have similar juice Brix levels, at least in ON (Table 2). Higher RS concentration in Icewines from BC was also reported by Cliff *et al.*² and Nurgel *et al.*³.

Ethanol concentration did not differ significantly across province, although the suggested lower concentration in BC wines compared to ON agrees with the trends observed in the data of Cliff *et al.*² and Nurgel *et al.*³. In contrast with the latter study where higher acetic acid concentrations were reported for BC Icewines, we found no differences between provinces for VA. Some differences are observed between provinces for SO₂ and SA concentrations, although we advise caution in interpreting this result given the sampling concern noted above. Given the low number of samples from BC and NS, we recommend future surveys incorporate greater representation from these provinces where possible.

Across the 1995-2000 vintages, Nurgel *et al.*³ reported differences for a number of Icewine parameters, including glucose, fructose, ethanol and ethyl acetate. In our study, JB (within ONT) varied with vintage, with juice from the 2001 vintage approximately 1.2 °Brix lower than the other four years, possibly reflecting warmer harvest and pressing temperatures that year. Interestingly, the lower juice Brix did not translate into clear differences compared to other vintages for either wine RS or ethanol concentration. Conversely, wines from 2000 had significantly higher RS than those from three of the other four years, and had the highest JB value. Ethanol and VA did not differ between vintages. Overall, a stylistic trend may cautiously be suggested towards higher ethanol and lower RS in more recent vintages.

Varietal and style differences: Vidal and Riesling - Canada's most predominant Icewine varieties¹ - do not differ in JB, wine RS or ethanol concentration. By contrast, the data of Cliff *et al.*² shows higher RS in BC Riesling than in ONT Vidal, and Nurgel *et al.*³ found higher glucose but not fructose concentrations in Riesling

compared to Vidal. Given the much larger sample size in the current study, we speculate that the higher RS previously reported in Riesling is due to sampling bias; province rather than variety is responsible for the higher concentration for Riesling. While not significant, the ethanol concentration for Vidal is 0.5% v/v higher than Riesling, the same trend observed in Cliff *et al.*² and Nurgel *et al.*³ and may also be attributed to provincial factors. The concentration of VA is lower in Vidal than for any other variety; differences in VA or acetic acid values have not previously been reported between Icewine varieties.

For the less common varieties, Gewurztraminer and Chardonnay stand out with high JB and VA, parameters that are generally positively correlated (Table 3). Cabernet Franc and Cabernet Sauvignon, the two dominant varieties used for red Icewine, have similar values for juice and wine analytes to Vidal and Riesling, although a trend towards higher ethanol concentration is suggested. Winter hardiness, high natural acidity, late ripening, tough skins and stems and reasonable resistance to disease have been identified as the most important characteristics of Icewine grape varieties¹, and largely explains the predominance of Vidal and Riesling. It remains to be determined how suitable and sustainable these less common varieties will be. The higher VA observed in oaked Icewines may be due to the increased potential for microbial activity that exists in barrel compared to stainless steel, and/or the trend of higher juice Brix for these wines compared to un-oaked samples (Table 2).

Conclusions

This study surveys a much larger sample of Icewines and varieties than previously reported on, and characterizes the unique composition of the style, particularly in regard to high RS and VA concentrations. Province, vintage, variety and style were all sources of variation for at least some of the components measured, highlighting the complexity and heterogeneity of Icewine. Some of the variation in composition previously ascribed in the literature to varietal differences may more likely be due to factors related to region-of-origin, particularly harvest/pressing temperatures and winery practices. To more fully understand the factors affecting Icewine composition and quality, further research needs to include analysis of pressing temperatures and juice composition across all viticultural areas, as well as assess the sensory qualities of the wine.

Acknowledgements

The Natural Science and Engineering Research Council of Canada is gratefully acknowledged for its financial support of this research. We also thank the following individuals for their contributions: Steve Cater and Dorina Brasoveanu (LCBO), Lynda Van Zuiden

(CCOVI), Dr. Debbie Inglis (Brock University) and Laurie MacDonald (VQAO).

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