

## PROGRESS REPORT

**Comment:** Funds do not come to us, but to OSU. Not in RMIS

**PROJECT NO.:** OSU-ARF 3808

**TITLE:** Identification of Compounds Contributing to 'Gala' Apple Flavor

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**PERIOD COVERED:** 1993-1997 **TERMINATING YEAR:** 1997

### ACCOMPLISHMENTS:

1. An analytical method was developed in 1994 and perfected in 1995 that combined gas chromatography using a flame ionization detector (GC/FID) with olfactometry (GC/O). Volatile compounds emitted by Gala apples were sampled for 6, 12 or 24 hours on charcoal (ORBO 32, SUPELCO) or Tenax traps, then eluted with carbon disulfide (CS<sub>2</sub>) and diethyl ether, respectively. Four panelists evaluated the GC effluents following the OSME method developed at Oregon State University. Charcoal eluted with CS<sub>2</sub> had a larger adsorbing capacity than Tenax, and when sampled for 24 hours, resulted in the largest amount of perceived peaks. Therefore, it was used for the rest of the study.
2. Volatile compounds were identified by matching retention time and mass-spectra with standards. Odor peaks were perceived for Gala by olfactometry (Table 1). Twenty five compounds were described as fruity, sweet, apple-like or like other fruit (i.e. pear, banana). There also were peaks with odors described as rubber, skunk, mushroom, anise, cucumber, watermelon, floral, tape, spicy, and perfumey. A total of 38 peaks were identified by GC/MS, among which 24 were odor-active. Fruity compounds were largely esters, one of the two anise

peaks was 4-allylanisole, and one of the mushroom peaks was hexyl tiglate. Rubber and skunk peaks were not detected by the FID and could not be identified.

3. Compounds with the highest odor intensity (bold characters, Table 1) were mixed in water in the same proportions as found in Gala headspace and compared to apples presented in 1-gal jars. A screening test design (Echip v.6.1.2) was used, with 16 variables and 8 replicates. Hexyl acetate, hexanal and butyl acetate contributed the most to Gala aroma. However, the results were only partially reproducible, explained by a large variation between apples. Additionally, we could not evaluate the impact of the compounds having a large odor intensity in the OSME method. A matching odor experiment will be repeated with essences of Gala apples to overcome variation between samples.
4. 'Gala' apples harvested 12 Sept. 1995 near Manson, WA were stored for 1, 2.5 and 5 months in RA, CA and a combination of both. Apple aroma was described by a trained panel. Individual volatile compounds were quantified by using peak relative area (FID detector) and odor intensity (panelist response). All but two compounds decreased significantly after 4 months in CA storage. 3-methyl 2-butenyl acetate (fruity) and a garlic peak increased in CA. However, the increase of those two compounds could not compensate the general decreased of fruity aroma perceived by the descriptive panel for CA stored fruit.

#### **RESULTS:**

Acetate esters, particularly butyl acetate and hexyl acetate, are the primary components of Gala fruity aroma. Additionally, 4-allylanisole is the spicy, anise seed character of Gala aroma. The aroma of mixtures of standards of these compounds in water were perceived by trained panelists to be similar to the aroma of intact Gala apples. Several sulfur compounds were perceived by trained panelists by using GC/O and

descriptive sensory analysis, however the chemical identity of these compounds is unknown. Concentrations of the primary components of Gala aroma decrease during CA storage and recovery in air storage after CA is insignificant.

Results from this project will enable 'Gala' volatile analysis to be simplified by focusing only on compounds significantly contributing to flavor. Identification of specific acetate esters as primary components of Gala aroma has been used to design and conduct experiments to enhance Gala ester content (project ARS 528, J. Mattheis, J. Fellman). Identification of compounds previously not known as contributors to 'Gala' flavor will provide the basis for continued study of 'Gala' response to CA and aroma enhancement in storage. Results will be transferred by journal articles, presentations at industry meetings and via the USDA, ARS, Tree Fruit Research Laboratory web site AT <http://www.tfrrl.ars.usda.gov>.

Table 1. Olfactometry peaks for Gala perceived by four panelists, corresponding chemical identity, presence in 1994 and 1995, and perceived intensity.

Peak	Descriptor	Compound	Perceived		
			1994	1995	intensity (0 - 15)
1	tea/garlic/leaves	2-methyl propyl acetate		no	X 2.23
2	<b>sweet fruity</b>	<b>methyl-2-methyl butyrate</b>		X	X 8.91
3	fruity <sup>b</sup>	propyl propanoate		X	X 0.00
4	<b>nail polish</b>	<b>butyl acetate</b>	X	X	10.27
5	skunk, rubber	no peak		X	X 8.96
6	<b>sweet, strawberry</b>	<b>ethyl-2-methyl butyrate</b>		X	X 6.16
7	<b>solventy</b>	<b>2-methyl butyl acetate</b>		X	X 11.03
8	oatmeal, skunky	no peak		no	X 2.14
9	fruity <sup>b</sup>	propyl butyrate		no	X 0.00
10	garlic <sup>d</sup>	no peak		X	X 0.67
11	<b>fruity, apple</b>	<b>butyl propanoate</b>		X	X 1.48
12	<b>gala</b>	<b>pentyl acetate</b>	X	X	7.21
13	fruity, sweet, solvent	3-methyl-2-butenyl acetate		X	X 0.35
14	<b>sweet, strawberry</b>	<b>propyl-2-methyl butyrate</b>		X	X 6.77
15	rubber	no peak		X	X 10.20
16	mushroom	no peak	no	X	2.89
17	grassy, green apple	3-methyl butyl propanoate		X	X 0.77
18	fruity, tape	6-methyl-5-hepten-2-one	X	X	3.29
19	rotten apple	butyl butyrate		X	X 2.26
20	solventy, gala	unk. <sup>e</sup>		X	X 3.10
21	apple + toast	unk.		no	X 7.00
22	<b>GALA, ripe, pear</b>	<b>hexyl acetate</b>		X	X 11.35
23	metallic, skunk	no peak		X	X 1.59
24	sweet, anise <sup>b</sup>	unk.		X	X 1.27
25	<b>fruity, apple</b>	<b>butyl-2-methyl butyrate</b>		X	X 5.58
26	<b>watermelon</b>	<b>pentyl butyrate isomer</b>		X	X 6.91
27	oatmeal or fruity <sup>b</sup>	unk.		X	X 0.00
28	dusty/musty	no peak		no	X 6.36
29	floral	unk.		X	X 1.88
30	apple	hexyl propanoate		X	X 2.99
31	cucumber	unk.		no	X 2.23
32	anise/spice	unk.	X	X	2.62
33	tape/fruity	unk.		no	X 1.66
34	<b>green apple</b>	<b>butyl hexanoate<sup>c</sup></b>		X	X 4.73
35	apple	hexyl butyrate <sup>c</sup>		X	X 2.23
36	<b>anise, licorice</b>	<b>4-allylanisole</b>		X	X 7.57

37	cucumber <sup>a, b</sup>	unk.		X	X	0.82
38	cat urine, mushroom	unk.		X	X	5.20
39	<b>apple, grapefruit</b>	<b>hexyl-2-methyl butyrate</b>		X	X	5.11
40	strong rubber	no peak		X	X	5.44
41	grape uice <sup>a</sup> unk.		X	X		1.68
42	tape	unk.		X	X	1.74
43	nutty, mushroom <sup>b</sup>	hexyl tiglate		X	X	0.74
44	tape or musty dirty	unk.		no	X	1.65
45	apple or tape <sup>b</sup>	unk.		no	X	0.87
46	<b>grape juice</b>	<b>damascenone</b>		X	X	6.18
47	fruity	unk.		X	X	1.58

a: perceived by one panelist only.

b: at or below odor threshold. Perceived sporadically.

c: peaks co-elute on the FID, but perceived separately by the panelists

d: mostly in CA-stored fruit

e: correspond to peaks detected by FID, but no match found in the NIST MS library