

ANALYSIS OF ACB'S AND VOLATILES
FROM COMMERCIALY PROCESSED
IRRADIATED FOODS

By

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For

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Testing Methods

Meat samples were obtained from Public Citizen. They consisted of uncooked control meat, Surebeam meat from two locations, New Generation meat patties and Dairy Queen cooked patties. For each of the raw meat samples, analysis was done both on raw meat and cooked meat. Patties with a similar thickness to the New Generation raw patties were made with the control and Surebeam meat, cooking on each side until the meat was thoroughly browned on each side in a skillet. Past experience has shown that grilling meat at very high temperature is problematic and can create pyrolysis products that interfere with many meat analyses. Grilling can also generate volatiles that may be chemically identical to the volatiles that are of interest to Public Citizen. All meat samples were homogenized before extraction of volatiles or ACBs.

Total Fat

The total fat was performed using a Paley bottle (Modified Babcock) using the standard USDA and AOAC procedure.

Volatiles

Volatiles were analyzed by an adaptation of the method of Champagne & Nawar where the volatiles were collected in a liquid nitrogen dewar condenser after distillation from the hamburger (100 gm) at up to 80 C. This method allows volatile components from fats and other components of meats to be measured. In this work, this method allows the concentration of components arising from fat that result from chemical modifications of the free radicals resulting from irradiation treatments. Unlike the procedure, the headspace volatiles from this extraction procedure being held at 40 C were injected into the gas chromatograph to identify low molecular weight compounds having high partial pressures at room temperature. In the second gas chromatographic injection, other less volatile compounds were partitioned from the collected condensate using sodium sulfate and pentane fractionation. It was not necessary to make a third separate fractionation with xylene to quantitate compounds having intermediate boiling points similar to pentane. For each of the compounds that were tracked a series of recovery mixes were used which measured known amounts of each target compound standard after fractionation to quantitate original levels of these compounds in the sample. We were able to identify all target compounds without interference from other compounds in the meat down to 1 ppb levels, which was at the limit of sensitivity using FID detection. The organic acids heptanoic acid and octanoic acids were easily seen in the second GC injection after partitioning without interference. Therefore, a separate methylation step was not necessary which allowed more reliable results.

For the low boiling point volatile compounds, a 1/8" X 6 ft alumina column was used with a temperature program of 50 F for 5 minutes, a temperature ramp of 5/min. to a maximum of 200 C. for 20 min. The higher boiling point compounds were separated using a 30-meter OV-1 capillary column. For the higher boiling point compounds, the pentane extract was concentrated to 0.5 ml on a Kuderna-Danish concentrator. All gas

chromatography results were performed with FID detection using methylene chloride as or cyclohexane as internal standards.

ACB (alkyl cyclobutanone) analysis

The extraction of the homogenized meat was performed with 3, 50 ml portions of hexane. The standards 2-dodecylcyclobutanone and 2-tetradecenylcyclobutanone were purchased from Fluka. The other ACBs found 2-octylcyclobutanone and 2-tetradecylcyclobutanone were not available from any supplier that we could find. In the recovery samples, a 1 ug and one other level of the standard was incorporated into control meat and looked at the levels after gas chromatography. A Florisil cleanup with deactivated Florisil was used (J. Food Prot. 53(11):1563-156). The fraction containing the ACB's was concentrated to 0.5 ml with a Kuderna-Danish concentrator. A 1 ul aliquot of this fraction, which contained 2 cyclohexyl cyclohexanone or benzyl alcohol, was used as the internal standard. The first analysis of the ACBs was done by GC-MS and the second replicate was run using GC-FID. Final results were calculated based upon recovery mix samples in the meat or per gm of fat.

RESULTS

The results for the volatile and ACB analysis follow. The volatile results are also supplied in excel format which may be easier to work with. The results do not show any volatiles that cause any concern over non-irradiated meat samples that we examined.

The ACB analyses also follow. There were some differences from expectation that have already been discussed as answers to questions of Mr. Peter Jenkins. One interesting findings was that the Dairy Queen ACBs were lower than expected. Levels of ACBs were also different in gamma-irradiated hamburger versus electron beam irradiated hamburger. Otherwise, the levels found are consistent with findings from others.

Percent Fat by Paley Bottle (AOAC and USDA)

Sample	Percent Fat	Correction Factor Per Control
Control Hamburger	6.5	1
Surebeam # 1	8.5	0.764706
Surebeam # 2	5.8	1.12069
New Generation	6.3	1.031746
Dairy Queen	22.7	0.286344

ACB RESULTS

	2-dOCB		2-dDCB		2-tDeDCB		2-tDCB		Fat
	per fat	per meat	per fat	per meat	per fat	per meat	per fat	per meat	
	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	
Control Raw	0.000	0.0000	0.000	0.0000	0.000	0.0000	0.000	0.0000	6.5% fat
	0.000	0.0000	0.000	0.0000	0.000	0.0000	0.000	0.0000	
Control Cooked	0.000	0.0000	0.000	0.0000	0.000	0.0000	0.000	0.0000	6.5% fat
	0.000	0.0000	0.000	0.0000	0.000	0.0000	0.000	0.0000	
Surebeam Raw #1	0.24	0.0139	0.720	0.0612	0.410	0.0349	0.1	0.0058	8.5% fat
	0.45	0.0261	0.780	0.0663	0.440	0.0374	0.096	0.0056	
Surebeam Cooked #1	0.56	0.0476	0.64	0.0544	0.073	0.0062	0.008	0.0007	8.5% fat
	0.21	0.0179	0.73	0.0621	0.101	0.0086	0.01	0.0009	
Surebeam Raw #2	0.41	0.0238	0.57	0.0331	0.14	0.0081	0.014	0.0008	5.8% fat
	0.38	0.0220	0.53	0.0307	0.087	0.0050	0.013	0.0008	
Surebeam Cooked #2	0.29	0.0168	0.51	0.0296	0.14	0.0081	0.034	0.0020	5.8% fat
	0.31	0.0180	0.48	0.0278	0.13	0.0075	0.03	0.0017	
New Generation Raw	0.36	0.0234	1.5	0.0975	0.06	0.0039	0.093	0.0054	6.3% fat
	0.4	0.0260	1.6	0.1040	0.054	0.0035	0.1	0.0058	
New Generation Cooked	0.41	0.0267	1.4	0.0910	0.027	0.0018	0.02	0.0012	6.3% fat
	0.38	0.0247	1.28	0.0832	0.025	0.0016	0.013	0.0008	
Dairy Queen #1	0.01	0.0020	0.081	0.0178	0.0015	0.0003	0.004	0.0002	22% fat
	0.01	0.0026	0.084	0.0185	0.0012	0.0003	0.004	0.0002	

Abbrev.	Compound	Determination
2-dOCB	2-octyl cyclobutanone	By Calculation
2-dDCB	2-dodecylcyclobutanone	By Standard
2-tDeDCB	2-tetradecenylcyclobutanone	By Standard
2-tDCB	2-tetradecylcyclobutanone	By Calculation

Volatiles found in Meat Samples (ppb)

	Raw Control #1		Raw Control #2	
	Sample #1	per gm Fat	Sample #2	per gm Fat
Acetaldehyde	0	0.0	0	0.0
Acetone	0	0.0	0	0.0
Acetonitrile	0	0.0	0	0.0
Benzene	0	0.0	0	0.0
Butane	0.04	0.6	0.02	0.3
Butanol	0	0.0	0	0.0
Butanone	0	0.0	0	0.0
Butene	0.01	0.2	0.07	1.1
Decyne	0	0.0	0	0.0
Ethane	0.1	1.5	0.1	1.5
Ethanol	0.3	4.6	0.3	4.6
Ethene	0	0.0	0	0.0
Ethylene	0.1	1.5	0.1	1.5
Ethyl mercaptan	0	0.0	0	0.0
Ethylbenzene	0	0.0	0	0.0
Heptanal	0	0.0	0	0.0
Heptane	0	0.0	0	0.0
Heptanoic Acid	0	0.0	0	0.0
Hexanal	0	0.0	0	0.0
Hexane	0	0.0	0	0.0
Hexene	0	0.0	0	0.0
Isopentane (2-Methylbutane)	0	0.0	0	0.0
Methane	0.2	3.1	0.2	3.1
Methanol	0.1	1.5	0.1	1.5
Methyl Disulfide	0	0.0	0	0.0
Methyl Ethyl Ketone	0	0.0	0	0.0
Methyl Sulfide	0	0.0	0	0.0
Methyl Vinyl Ketone	0	0.0	0	0.0
Octanal	0	0.0	0	0.0
Octane	0	0.0	0	0.0
Octanoic acid	0	0.0	0	0.0
Pentanal	0	0.0	0	0.0
Pentane	1.6	24.6	1.4	21.5
Pentent	0.2	3.1	0.1	1.5
Propane	0.18	2.8	0.25	3.8
Propanol	0	0.0	0	0.0
Propene	0.2	3.1	0.1	1.5
Propylbenzene	0	0.0	0	0.0
Tetrachloroethylene	0	0.0	0	0.0
Trichloroethylene	0	0.0	0	0.0
Toluene	0	0.0	0	0.0
Xylene	0	0.0	0	0.0

Volatiles found in Meat Samples (ppb)

	Cooked Control #1		Cooked Control #2	
	Sample #1	per gm Fat	Sample #2	per gm Fat
Acetaldehyde	0	0.0	0	0.0
Acetone	0	0.0	0	0.0
Acetonitrile	0	0.0	0	0.0
Benzene	0	0.0	0	0.0
Butane	0.01	0.2	0.01	0.2
Butanol	0	0.0	0	0.0
Butanone	0	0.0	0	0.0
Butene	0.05	0.8	0.06	0.9
Decyne	0	0.0	0	0.0
Ethane	0.1	1.5	0.1	1.5
Ethanol	0.5	7.7	0.2	3.1
Ethene	0	0.0	0	0.0
Ethylene	0	0.0	0	0.0
Ethyl mercaptan	0	0.0	0	0.0
Ethylbenzene	0	0.0	0	0.0
Heptanal	0	0.0	0	0.0
Heptane	0	0.0	0	0.0
Heptanoic Acid	0	0.0	0	0.0
Hexanal	0	0.0	0	0.0
Hexane	0	0.0	0	0.0
Hexene	0	0.0	0	0.0
Isopentane (2-Methylbutane)	0	0.0	0	0.0
Methane	0.1	1.5	0.1	1.5
Methanol	0.1	1.5	0.1	1.5
Methyl Disulfide	0	0.0	0	0.0
Methyl Ethyl Ketone	0	0.0	0	0.0
Methyl Sulfide	0	0.0	0	0.0
Methyl Vinyl Ketone	0	0.0	0	0.0
Octanal	0	0.0	0	0.0
Octane	0	0.0	0	0.0
Octanoic acid	0	0.0	0	0.0
Pentanal	0	0.0	0	0.0
Pentane	1	15.4	0.6	9.2
Pentent	0.1	1.5	0.1	1.5
Propane	0.21	3.2	0.05	0.8
Propanol	0	0.0	0	0.0
Propene	0.1	1.5	0.1	1.5
Propylbenzene	0	0.0	0	0.0
Tetrachloroethylene	0	0.0	0	0.0
Trichloroethylene	0	0.0	0	0.0
Toluene	0	0.0	0	0.0
Xylene	0	0.0	0	0.0

Volatiles found in Meat Samples (ppb)

	Raw Surebeam #1		Raw Surebeam #1	
	Sample #1	per gm Fat	Sample #2	per gm Fat
Acetaldehyde	0	0.0	0	0.0
Acetone	0	0.0	0	0.0
Acetonitrile	0	0.0	0	0.0
Benzene	0	0.0	0	0.0
Butane	0	0.0	0	0.0
Butanol	0	0.0	0	0.0
Butanone	0	0.0	0	0.0
Butene	0	0.0	0	0.0
Decyne	0	0.0	0	0.0
Ethane	0	0.0	0	0.0
Ethanol	0	0.0	0	0.0
Ethene	0	0.0	0	0.0
Ethylene	0.1	1.2	0	0.0
Ethyl mercaptan	0	0.0	0	0.0
Ethylbenzene	0	0.0	0	0.0
Heptanal	0	0.0	0	0.0
Heptane	0	0.0	0	0.0
Heptanoic Acid	0	0.0	0	0.0
Hexanal	0	0.0	0	0.0
Hexane	0	0.0	0	0.0
Hexene	0	0.0	0	0.0
Isopentane (2-Methylbutane)	0	0.0	0	0.0
Methane	0.1	1.2	0	0.0
Methanol	0	0.0	0	0.0
Methyl Disulfide	0	0.0	0	0.0
Methyl Ethyl Ketone	0	0.0	0	0.0
Methyl Sulfide	0	0.0	0	0.0
Methyl Vinyl Ketone	0	0.0	0	0.0
Octanal	0	0.0	0	0.0
Octane	0	0.0	0	0.0
Octanoic acid	0	0.0	0	0.0
Pentanal	0	0.0	0	0.0
Pentane	0.4	4.7	2	23.5
Pentent	0	0.0	0	0.0
Propane	0.04	0.5	0	0.0
Propanol	0	0.0	0	0.0
Propene	0	0.0	0	0.0
Propylbenzene	0	0.0	0	0.0
Tetrachloroethylene	0	0.0	0	0.0
Trichloroethylene	0	0.0	0	0.0
Toluene	0	0.0	0	0.0
Xylene	0	0.0	0	0.0

Volatiles found in Meat Samples (ppb)

	Cooked Surebeam #1		Cooked Surebeam	
	Sample #1	per gm Fat	Sample #2	per gm Fat
Acetaldehyde	0	0.0	0	0.0
Acetone	0	0.0	0	0.0
Acetonitrile	0	0.0	0	0.0
Benzene	0	0.0	0	0.0
Butane	0	0.0	0	0.0
Butanol	0	0.0	0	0.0
Butanone	0	0.0	0	0.0
Butene	0	0.0	0	0.0
Decyne	0	0.0	0	0.0
Ethane	0	0.0	0	0.0
Ethanol	0.1	1.2	0.3	3.5
Ethene	0	0.0	0	0.0
Ethylene	0.1	1.2	0	0.0
Ethyl mercaptan	0	0.0	0	0.0
Ethylbenzene	0	0.0	0	0.0
Heptanal	0	0.0	0	0.0
Heptane	0	0.0	0	0.0
Heptanoic Acid	0	0.0	0	0.0
Hexanal	0	0.0	0	0.0
Hexane	0	0.0	0	0.0
Hexene	0	0.0	0	0.0
Isopentane (2-Methylbutane)	0	0.0	0	0.0
Methane	0.2	2.4	0	0.0
Methanol	0.2	2.4	0	0.0
Methyl Disulfide	0	0.0	0	0.0
Methyl Ethyl Ketone	0	0.0	0	0.0
Methyl Sulfide	0	0.0	0	0.0
Methyl Vinyl Ketone	0	0.0	0	0.0
Octanal	0	0.0	0	0.0
Octane	0	0.0	0	0.0
Octanoic acid	0	0.0	0	0.0
Pentanal	0	0.0	0	0.0
Pentane	0.2	2.4	0.2	2.4
Pentent	0	0.0	0	0.0
Propane	0.16	1.9	0.03	0.4
Propanol	0	0.0	0	0.0
Propene	0	0.0	0	0.0
Propylbenzene	0	0.0	0	0.0
Tetrachloroethylene	0	0.0	0	0.0
Trichloroethylene	0	0.0	0	0.0
Toluene	0	0.0	0	0.0
Xylene	0	0.0	0	0.0

Volatiles found in Meat Samples (ppb)

Sample	Raw Surebeam 2		Raw Surebeam 2	
	#1	per gm Fat	#2	per gm Fat
Acetaldehyde	0	0.0	0	0.0
Acetone	0	0.0	0	0.0
Acetonitrile	0	0.0	0	0.0
Benzene	0	0.0	0	0.0
Butane	0	0.0	0	0.0
Butanol	0	0.0	0	0.0
Butanone	0	0.0	0	0.0
Butene	0	0.0	0	0.0
Decyne	0	0.0	0	0.0
Ethane	0.1	1.7	0.5	8.6
Ethanol	0	0.0	0	0.0
Ethene	0	0.0	0	0.0
Ethylene	0	0.0	0	0.0
Ethyl mercaptan	0	0.0	0	0.0
Ethylbenzene	0	0.0	0	0.0
Heptanal	0	0.0	0	0.0
Heptane	0	0.0	0	0.0
Heptanoic Acid	0	0.0	0	0.0
Hexanal	0	0.0	0	0.0
Hexane	0	0.0	0	0.0
Hexene	0	0.0	0	0.0
Isopentane (2-Methylbutane)	0	0.0	0	0.0
Methane	0.1	1.7	0.1	1.7
Methanol	0	0.0	0	0.0
Methyl Disulfide	0	0.0	0	0.0
Methyl Ethyl Ketone	0	0.0	0	0.0
Methyl Sulfide	0	0.0	0	0.0
Methyl Vinyl Ketone	0	0.0	0	0.0
Octanal	0	0.0	0	0.0
Octane	0	0.0	0	0.0
Octanoic acid	0	0.0	0	0.0
Pentanal	0	0.0	0	0.0
Pentane	0.1	1.7	0	0.0
Pentent	0	0.0	0	0.0
Propane	0.10	1.7	0.08	1.4
Propanol	0	0.0	0	0.0
Propene	0	0.0	0	0.0
Propylbenzene	0	0.0	0	0.0
Tetrachloroethylene	0	0.0	0	0.0
Trichloroethylene	0	0.0	0	0.0
Toluene	0	0.0	0	0.0
Xylene	0	0.0	0	0.0

Volatiles found in Meat Samples (ppb)

	Cooked Surebeam 2		Cooked Surebeam 2	
	Sample #1	per gm Fat	Sample #2	per gm Fat
Acetaldehyde	0	0.0	0	0.0
Acetone	0	0.0	0	0.0
Acetonitrile	0	0.0	0	0.0
Benzene	0	0.0	0	0.0
Butane	0	0.0	0	0.0
Butanol	0	0.0	0	0.0
Butanone	0	0.0	0	0.0
Butene	0	0.0	0	0.0
Decyne	0	0.0	0	0.0
Ethane	0.5	8.6	1.5	25.9
Ethanol	0	0.0	0	0.0
Ethene	0	0.0	0	0.0
Ethylene	0	0.0	0	0.0
Ethyl mercaptan	0	0.0	0	0.0
Ethylbenzene	0	0.0	0	0.0
Heptanal	0	0.0	0	0.0
Heptane	0	0.0	0	0.0
Heptanoic Acid	0	0.0	0	0.0
Hexanal	0	0.0	0	0.0
Hexane	0	0.0	0	0.0
Hexene	0	0.0	0	0.0
Isopentane (2-Methylbutane)	0	0.0	0	0.0
Methane	0.2	3.4	0.3	5.2
Methanol	0	0.0	0	0.0
Methyl Disulfide	0	0.0	0	0.0
Methyl Ethyl Ketone	0	0.0	0	0.0
Methyl Sulfide	0	0.0	0	0.0
Methyl Vinyl Ketone	0	0.0	0	0.0
Octanal	0	0.0	0	0.0
Octane	0	0.0	0	0.0
Octanoic acid	0	0.0	0	0.0
Pentanal	0	0.0	0	0.0
Pentane	0.2	3.4	0.4	6.9
Pentent	0	0.0	0	0.0
Propane	0.19	3.3	0.28	4.8
Propanol	0	0.0	0	0.0
Propene	0	0.0	0	0.0
Propylbenzene	0	0.0	0	0.0
Tetrachloroethylene	0	0.0	0	0.0
Trichloroethylene	0	0.0	0	0.0
Toluene	0	0.0	0	0.0

Xylene 0 0.0 0 0.0
 Volatiles found in Meat Samples (ppb)

	Raw New Generation Sample	per gm Fat	Cooked New Generation Sample	per gm Fat
Acetaldehyde	0	0.0	0	0.0
Acetone	0	0.0	0	0.0
Acetonitrile	0	0.0	0	0.0
Benzene	0	0.0	0	0.0
Butane	0	0.0	0	0.0
Butanol	0	0.0	0	0.0
Butanone	0	0.0	0	0.0
Butene	0	0.0	0	0.0
Decyne	0	0.0	0	0.0
Ethane	0.5	7.9	0.9	14.3
Ethanol	1	15.9	1.5	23.8
Ethene		0.0		0.0
Ethylene	0.2	3.2	0	0.0
Ethyl mercaptan	0	0.0	0	0.0
Ethylbenzene	0	0.0	0	0.0
Heptanal	0	0.0	0	0.0
Heptane	0	0.0	0	0.0
Heptanoic Acid	0	0.0	0	0.0
Hexanal	0	0.0	0	0.0
Hexane	0	0.0	0	0.0
Hexene	0	0.0	0	0.0
Isopentane (2-Methylbutane)	0	0.0	0	0.0
Methane	0.3	4.8	0.3	4.8
Methanol	0.9	14.3	1.9	30.0
Methyl Disulfide	0	0.0	0	0.0
Methyl Ethyl Ketone	0	0.0	0	0.0
Methyl Sulfide	0	0.0	0	0.0
Methyl Vinyl Ketone	0	0.0	0	0.0
Octanal	0	0.0	0	0.0
Octane	0	0.0	0	0.0
Octanoic acid	0	0.0	0	0.0
Pentanal	0	0.0	0	0.0
Pentane	0.3	4.8	0.2	3.2
Pentent	0	0.0	0	0.0
Propane	0.44	7.0	0.34	5.2
Propanol	0	0.0	0	0.0
Propene	0	0.0	0	0.0
Propylbenzene	0	0.0	0	0.0
Tetrachloroethylene	0	0.0	0	0.0
Trichloroethylene	0	0.0	0	0.0
Toluene	0	0.0	0	0.0
Xylene	0	0.0	0	0.0

Volatiles found in Meat Samples (ppb)

	Raw Dairy Queen		Cooked Dairy Queen	
	Sample	per gm Fat	Sample	per gm Fat
Acetaldehyde	0	0.0	0	0.0
Acetone	0	0.0	0	0.0
Acetonitrile	0	0.0	0	0.0
Benzene	0	0.0	0	0.0
Butane	0	0.0	0	0.0
Butanol	0	0.0	0	0.0
Butanone	0	0.0	0	0.0
Butene	0	0.0	0	0.0
Decyne	0	0.0	0	0.0
Ethane	0	0.0	0	0.0
Ethanol	2.8	127.3	3.6	163.6
Ethene		0.0		0.0
Ethylene	0	0.0	0	0.0
Ethyl mercaptan	0	0.0	0	0.0
Ethylbenzene	0	0.0	0	0.0
Heptanal	0	0.0	0	0.0
Heptane	0	0.0	0	0.0
Heptanoic Acid	0	0.0	0	0.0
Hexanal	0	0.0	0	0.0
Hexane	0	0.0	0	0.0
Hexene	0	0.0	0	0.0
Isopentane (2-Methylbutane)	0	0.0	0	0.0
Methane	0.1	4.5	0.1	4.5
Methanol	0	0.0	0	0.0
Methyl Disulfide	0	0.0	0	0.0
Methyl Ethyl Ketone	0	0.0	0	0.0
Methyl Sulfide	0	0.0	0	0.0
Methyl Vinyl Ketone	0	0.0	0	0.0
Octanal	0	0.0	0	0.0
Octane	0	0.0	0	0.0
Octanoic acid	0	0.0	0	0.0
Pentanal	0	0.0	0	0.0
Pentane	0	0.0	0	0.0
Pentent	0	0.0	0	0.0
Propane	0.98	44.5	0.97	44.1
Propanol	0	0.0	0	0.0
Propene	0	0.0	0	0.0
Propylbenzene	0	0.0	0	0.0
Tetrachloroethylene	0	0.0	0	0.0
Trichloroethylene	0	0.0	0	0.0
Toluene	0	0.0	0	0.0
Xylene	0	0.0	0	0.0