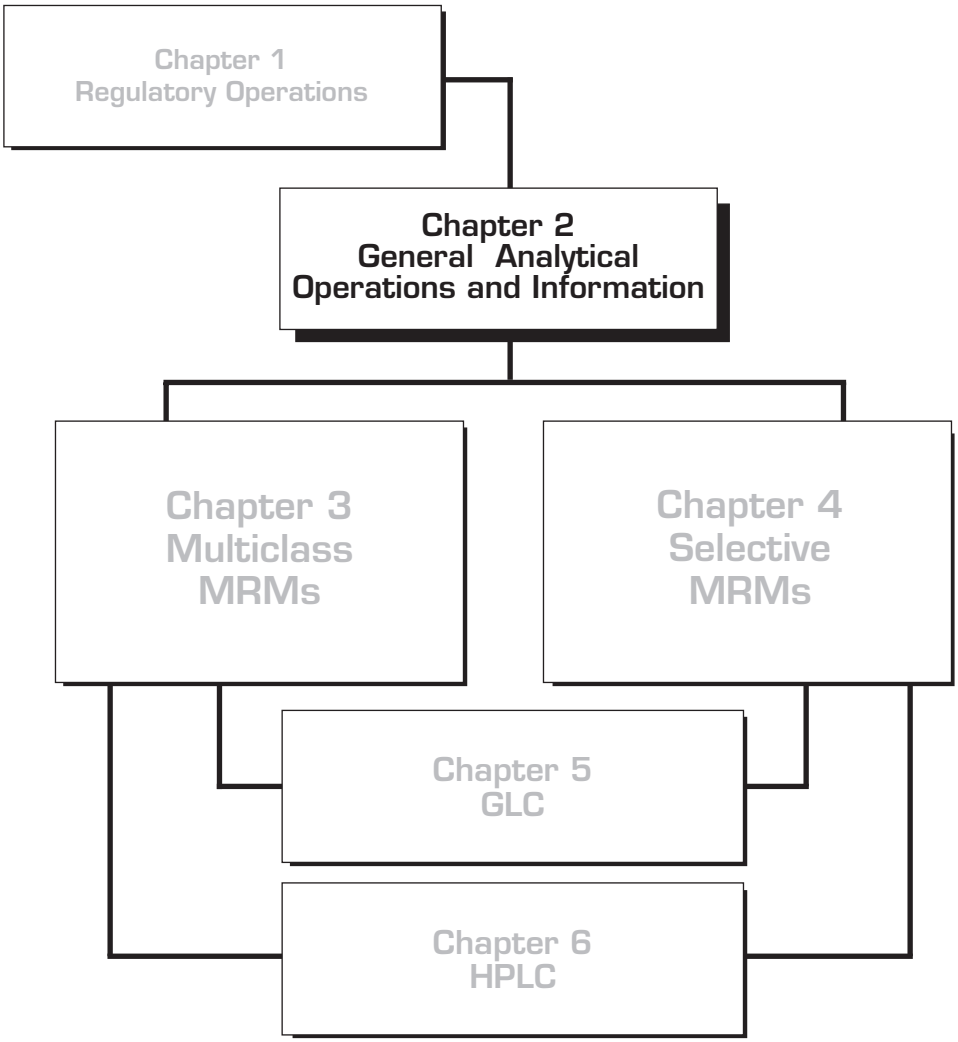

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201: PERCENTAGE FAT, WATER, AND SUGARS IN FOODS

Methods in Chapters 3 and 4 are usually designated as applicable to either fatty (>2%) or nonfatty (<2%) foods. In addition, some methods for nonfatty foods include alternative extraction steps, choice of which is dictated by the percentage water or sugars in the product. To facilitate proper application of these methods, this table provides percentage fat, water, and sugars for raw agricultural commodities and some processed foods.

Data were obtained from the following sources:

USDA Nutrient Data Base for Standard Reference, Release 8 and Release 9, U.S. Department of Agriculture, Washington, DC

Home Economics Research Report No. 48, "Sugar Content of Selected Foods: Individual and Total Sugars," Tables 1, 3, and 4, Stock No. 001-000-04515-8, Government Printing Office, Washington, DC 20402; obtained by download from bulletin board

Pehrsson, P. (Jan. 1994), private communication

Smith, J.S., *et al.* (1986) *J. Food Sci.* **51**, 1373-1375

Notes on the information in this table:

For the convenience of FDA field personnel, Office of Regulatory Affairs (ORA) product codes are included for most commodities, and commodities are grouped by the same categories used to create the codes. (Product codes are those used in the FDA reporting system; these are not the same codes used by USDA to identify commodities in the Nutrient Data Base.) Within each subcategory, items are sorted alphabetically by name of the commodity.

Names of commodities, including abbreviations, are those used by USDA Nutrient Data Base.

Percentage sugars represents the sum of one or more mono- and disaccharides. Data are not available for all commodities, but the table will be updated in the future as data become available.

For some commodities, sum of percent fat, water, and sugars may exceed 100%; this is caused by the fact that data were developed at different times on different samples.

Product Code	Commodity	% Fat	% Water	% Sugars
02: GRAINS				
A: whole grain				
02A-02	barley, pearled, raw	1.16	10.09	*
02A-99	buckwheat groats, roasted, dry	2.71	8.41	*
02A-01	corn, dry	2.08	10	*
02A-99	couscous, dry	0.64	8.56	*
02A-03	oat bran, raw	7.03	6.55	1.4
02A-03	oats	6.9	8.22	*
02A-04	popcorn: unpopped	4.7	9.8	*
02A-07	rye	2.5	10.95	*
02A-08	sorghum	3.3	9.2	*
02A-06	wild rice, raw	1.08	7.76	2.5
B: corn meal & milled corn products				
02B-01	corn flour, masa, enriched	3.78	9.03	*
02B-01	corn flour, whole-grain, white	3.86	10.91	*
02B-01	corn flour, whole-grain, yellow	3.86	10.91	*
C: rice flour				
02C-01	rice flour, brown	2.78	11.97	*
02C-01	rice flour, white	1.42	11.89	1
D: processed rice & milled rice products				
02D-99	rice bran, crude	20.85	6.13	0.9
02D-01	rice, brown, long-grain, raw	2.92	10.37	0.7
02D-01	rice, brown, medium-grain, raw	2.68	12.37	0.7
02D-10	rice, white, glutinous, raw	0.55	10.46	*
02D-03	rice, white, long-grain, parboiled, dry, enriched	0.56	10.16	*
02D-03	rice, white, long-grain, precooked or instant, enriched, dry	0.29	8.14	*
02D-03	rice, white, long-grain, regular, raw, enriched	0.66	11.62	0.5
02D-02	rice, white, medium-grain, cooked, enriched	0.21	68.61	0.2
02D-03	rice, white, medium-grain, raw, enriched	0.58	12.89	0.5
02D-02	rice, white, medium-grain, raw, unenriched	0.58	12.89	0.5
02D-03	rice, white, short-grain, raw, enriched	0.52	13.29	*
F: milled wheat product				
02F-04	semolina, enriched	1.05	12.67	*
G: other flour/milled products				
02G-02	buckwheat	3.4	9.75	*
02G-02	buckwheat flour, whole-groat	3.1	11.15	*
02G-10	rye flour, dark	2.69	11.07	3.5
02G-10	rye flour, light	1.36	8.78	3.5
02G-10	rye flour, medium	1.77	9.85	3.5

* Percent sugars not available.

Product Code	Commodity	% Fat	% Water	% Sugars
H: starch products				
02H-99	arrowroot flour	0.1	11.37	*
02H-02	cornstarch	0.05	8.32	*
09: DAIRY				
A: butter products				
09A-01	butter, wo/salt	81.11	17.94	*
09A-01	butter: w/salt	81.11	15.87	*
09A-01	butter: whipped	81.11	15.87	*
C: milk/cream				
09C-07	cream: fluid, half & half, cream and milk	11.5	80.57	*
09C-13	cream: fluid, heavy whipping	37	57.71	2.8
09C-13	cream: fluid, light whipping	30.91	63.5	2.8
09C-13	cream: fluid, light, coffee or table	19.31	73.75	*
09C-13	cream: fluid, medium, 25% fat	25	68.5	*
09C-12	cream: sour half & half, cultured	12	80.14	*
09C-04	eggnog	7.48	74.37	*
09C-03	milk: cow, cnd, condensed, sweetened	8.7	27.16	*
09C-03	milk: cow, cnd, evaporated, unsweetened, w/added Vit. A	7.56	74.04	*
09C-16	milk: cow, dry, whole	26.71	2.47	35.9
09C-09	milk: cow, whole, past and raw, fluid, 3.3% fat	3.34	87.99	4.9
09C-09	milk: cow, whole, past and raw, fluid, 3.7% fat	3.66	87.69	4.9
09C-12	sour cream	20.96	70.95	*
D: low fat milk product				
09D-03	milk: cow, cnd, evaporated, skim	0.2	79.4	*
09D-16	milk: cow, dry, skim, calcium reduced	0.2	4.9	*
09D-16	milk: cow, dry, skim, nonfat solids, instant, w/added Vit. A	0.72	3.96	50.4
09D-09	milk: cow, lowfat, past & raw, fluid, 1% fat	1.06	90.08	*
09D-09	milk: cow, lowfat, past & raw, fluid, 2% fat	1.92	89.21	*
E: non-fat milk product				
09E-01	milk: buttermilk, fluid, cultured, from skim milk	0.88	90.13	4.8
09E-16	milk: cow, dry, skim, nonfat solids, regular, w/added Vit. A	0.77	3.16	*
09E-11	milk: cow, skim, past & raw, fluid, w/added Vit. A	0.18	90.8	4.9
12: CHEESE				
A: standard cheese				
12A-02	cheese: natural, blue	28.74	42.41	*
12A-03	cheese: natural, brick	29.68	41.11	*
12A-56	cheese: natural, brie	27.68	48.42	*
12A-56	cheese: natural, camembert, domestic	24.26	51.8	*
12A-57	cheese: natural, caraway	29.2	39.28	*
12A-05	cheese: natural, cheddar, American domestic	33.14	36.75	1.8
12A-06	cheese: natural, colby	32.11	38.2	*

Product Code	Commodity	% Fat	% Water	% Sugars
12A-11	cheese: natural, cottage, creamed, large or small curd	4.51	78.96	0.6
12A-62	cheese: natural, cottage, low fat, 1% fat	1.02	82.48	*
12A-62	cheese: natural, cottage, low fat, 2% fat	1.93	79.31	*
12A-09	cheese: natural, cottage, uncreamed, dry, large or small curd	0.42	79.77	*
12A-10	cheese: natural, cream	34.87	53.75	1.7
12A-12	cheese: natural, edam	27.8	41.56	*
12A-16	cheese: natural, gouda	27.44	41.46	*
12A-18	cheese: natural, gruyere	32.34	33.19	*
12A-37	cheese: natural, limburger	27.25	48.42	*
12A-38	cheese: natural, monterey	30.28	41.01	*
12A-39	cheese: natural, muenster	30.04	41.77	*
12A-40	cheese: natural, neufchatel	23.43	62.21	1
12A-42	cheese: natural, Parmesan, grated	30.02	17.66	*
12A-52	cheese: natural, port de salut	28.2	45.45	*
12A-44	cheese: natural, provolone	26.62	40.95	*
12A-47	cheese: natural, romano	26.94	30.91	*
12A-49	cheese: natural, Roquefort	30.64	39.38	*
12A-60	cheese: natural, Swiss, domestic	27.45	37.21	1.2
12A-52	cheese: natural, tilsit, whole milk	25.98	42.86	*
B: standard cheese products				
12B-01	cheese food: cold pack, American	24.46	43.12	*
12B-09	cheese food: pasteurized processed, Swiss	24.14	43.67	*
12B-09	cheese: pasteurized processed, American, w/di Na phos	31.25	39.16	*
12B-13	cheese: pasteurized processed, Swiss, w/di Na phos	25.01	42.31	*
C: non-standard cheese products				
12C-99	cheese: natural, cheshire	30.6	37.65	*
12C-12	cheese: natural, feta	21.28	55.22	*
12C-04	cheese: natural, fontina	31.14	37.92	*
12C-99	cheese: natural, gjetost	29.51	13.44	*
12C-06	cheese: natural, mozzarella, part skim milk	15.92	53.78	*
12C-06	cheese: natural, mozzarella, part skim milk, low moisture	17.12	48.57	*
12C-06	cheese: natural, mozzarella, whole milk	21.6	54.14	*
12C-06	cheese: natural, mozzarella, whole milk, low moisture	24.64	48.38	*
12C-11	cheese: natural, ricotta, part skim milk	7.91	74.41	1.4
12C-11	cheese: natural, ricotta, whole milk	12.98	71.7	1.5
13: ICE CREAM, ETC.				
A: ice cream				
13A-02	ice cream: French, vanilla, soft serve	13.02	59.76	*
13A-03	ice cream: vanilla, regular, appx 10% fat	10.77	60.8	17.5
13A-02	ice cream: vanilla, rich, appx 16% fat	16	58.87	17.5
C: ice milk				
13C-01	ice milk: vanilla, hardened	4.3	68.62	*

Product Code	Commodity	% Fat	% Water	% Sugars
13C-99	ice milk: vanilla, soft serve	2.64	69.64	*
D: sherbet				
13D-01	sherbet: orange	1.98	66.07	*
14: MILK PRODUCTS				
B: imitation milk products				
14B-99	cream substitute: nondairy, liquid, w/hydr veg oil & soy protein	9.97	77.27	*
14B-99	cream substitute: nondairy, liquid, w/lauric acid oil & Na casn	9.97	77.27	*
14B-06	cream substitute: nondairy, powdered	35.48	2.21	*
15: EGGS AND EGG PRODUCTS				
A: shell eggs				
15A-01	eggs: chicken, white, raw, fresh and frozen	0	87.81	*
15A-01	eggs: chicken, whole, raw, fresh, and frozen	10.02	75.33	*
15A-03	eggs: duck, whole, fresh, raw	13.77	70.83	*
15A-99	eggs: goose, whole, fresh, raw	13.27	70.43	*
B: shelled egg products				
15B-02	eggs: chicken, yolk, dried	61.28	4.65	*
15B-02	eggs: chicken, yolk, raw, fresh	30.87	48.81	*
15B-02	eggs: chicken, yolk, raw, frozen	26.01	55	*
E: imitation/substitute egg products				
15E-03	egg substitute: frozen	11.11	73.1	*
15E-99	egg substitute: liquid	3.31	82.75	*
15E-99	egg substitute: powder	13	3.86	*
16: FISH				
A: fish				
16A-01	fish/shellfish: anchovy, European, raw	4.84	73.37	0.0
16A-03	fish/shellfish: bass, freshwater, mixed species, raw	3.69	75.66	0.0
16A-03	fish/shellfish: bass, striped, raw	2.33	79.22	0.0
16A-05	fish/shellfish: bluefish, raw	4.24	70.86	0.0
16A-99	fish/shellfish: burbot, raw	0.81	79.26	0.0
16A-08	fish/shellfish: butterfish, raw	8.02	74.13	0.0
16A-09	fish/shellfish: carp, raw	5.6	76.31	0.0
16A-10	fish/shellfish: catfish, channel, raw	4.26	76.39	0.0
16A-48	fish/shellfish: cisco, raw	1.91	78.93	0.0
16A-12	fish/shellfish: cod, Atlantic, raw	0.67	81.22	0.0
16A-12	fish/shellfish: cod, Pacific, raw	0.63	81.28	0.0
16A-13	fish/shellfish: croaker, Atlantic, raw	3.17	78.03	0.0
16A-14	fish/shellfish: cusk, raw	0.69	76.35	0.0
16A-51	fish/shellfish: dolphinfish, raw	0.7	77.55	0.0
16A-13	fish/shellfish: drum, freshwater, raw	4.93	77.33	0.0
16A-15	fish/shellfish: eel, mixed species, raw	11.66	68.26	0.0

Product Code	Commodity	% Fat	% Water	% Sugars
16A-16	fish/shellfish: flatfish (flounder and sole species), raw	1.19	79.06	0.0
16A-17	fish/shellfish: grouper, mixed species, raw	1.02	79.22	0.0
16A-18	fish/shellfish: haddock, raw	0.72	79.92	0.0
16A-20	fish/shellfish: halibut, Atlantic and Pacific, raw	2.29	77.92	0.0
16A-20	fish/shellfish: halibut, Greenland, raw	13.84	70.27	0.0
16A-21	fish/shellfish: herring, Atlantic, raw	9.04	72.05	0.0
16A-21	fish/shellfish: herring, Pacific, raw	13.88	71.52	0.0
16A-99	fish/shellfish: ling, raw	0.64	79.63	0.0
16A-99	fish/shellfish: lingcod, raw	1.06	81.03	0.0
16A-22	fish/shellfish: mackerel, Atlantic, raw	13.89	63.55	0.0
16A-22	fish/shellfish: mackerel, king, raw	2	75.85	0.0
16A-22	fish/shellfish: mackerel, Pacific and jack, mixed species, raw	7.89	70.15	0.0
16A-22	fish/shellfish: mackerel, Spanish, raw	6.3	71.67	0.0
16A-53	fish/shellfish: milkfish, raw	6.73	70.85	0.0
16A-99	fish/shellfish: monkfish, raw	1.52	83.24	0.0
16A-24	fish/shellfish: ocean perch, Atlantic, raw	1.63	78.7	0.0
16A-25	fish/shellfish: pike, northern, raw	0.69	78.92	0.0
16A-25	fish/shellfish: pike, walleye, raw	1.22	79.31	0.0
16A-28	fish/shellfish: pollock, Atlantic, raw	0.98	78.18	0.0
16A-28	fish/shellfish: pollock, walleye, raw	0.8	81.56	0.0
16A-29	fish/shellfish: pompano, Florida, raw	9.47	71.12	0.0
16A-99	fish/shellfish: pout, ocean, raw	0.91	81.36	0.0
16A-24	fish/shellfish: rockfish, Pacific, mixed species, raw	1.57	79.26	0.0
16A-50	fish/shellfish: roughy, orange, raw	7	75.9	0.0
16A-31	fish/shellfish: sablefish, raw	15.3	71.02	0.0
16A-32	fish/shellfish: salmon, Atlantic, raw	6.34	68.5	0.0
16A-32	fish/shellfish: salmon, chinook, raw	10.44	73.17	0.0
16A-32	fish/shellfish: salmon, chum, raw	3.77	75.38	0.0
16A-32	fish/shellfish: salmon, coho, raw	5.95	72.63	0.0
16A-32	fish/shellfish: salmon, pink, raw	3.45	76.35	0.0
16A-32	fish/shellfish: salmon, sockeye, raw	8.56	70.24	0.0
16A-99	fish/shellfish: scup, raw	2.73	75.37	0.0
16A-04	fish/shellfish: sea bass, mixed species, raw	2	78.27	0.0
16A-47	fish/shellfish: seatrout, mixed species, raw	3.61	78.09	0.0
16A-21	fish/shellfish: shad, American, raw	13.77	68.19	0.0
16A-35	fish/shellfish: shark, mixed species, raw	4.51	73.58	0.0
16A-99	fish/shellfish: sheepshead, raw	2.41	77.97	0.0
16A-36	fish/shellfish: smelt, rainbow, raw	2.42	78.77	0.0
16A-99	fish/shellfish: snapper, mixed species, raw	1.34	76.87	0.0
16A-39	fish/shellfish: spot, raw	4.9	75.95	0.0
16A-40	fish/shellfish: sturgeon, mixed species, raw	4.04	76.55	0.0
16A-41	fish/shellfish: sucker, white, raw	2.32	79.71	0.0
16A-99	fish/shellfish: sunfish, pumpkinseed, raw	0.7	79.5	0.0
16A-42	fish/shellfish: swordfish, raw	4.01	75.62	0.0
16A-99	fish/shellfish: tilefish, raw	2.31	78.9	0.0
16A-44	fish/shellfish: trout, mixed species, raw	6.61	71.42	0.0
16A-44	fish/shellfish: trout, rainbow, raw	3.36	71.48	0.0

Product Code	Commodity	% Fat	% Water	% Sugars
16A-45	fish/shellfish: tuna, fresh, bluefin, raw	4.9	68.09	0.0
16A-45	fish/shellfish: tuna, fresh, skipjack, raw	1.01	70.58	0.0
16A-45	fish/shellfish: tuna, fresh, yellowfin, raw	0.95	70.99	0.0
16A-46	fish/shellfish: turbot, European, raw	2.95	76.95	0.0
16A-48	fish/shellfish: whitefish, mixed species, raw	5.86	72.77	0.0
16A-49	fish/shellfish: whiting, mixed species, raw	1.31	80.27	0.0
16A-99	fish/shellfish: wolffish, Atlantic, raw	2.39	79.9	0.0
16A-99	fish/shellfish: yellowtail, mixed species, raw	5.24	74.52	0.0
16A-02	fish: barracuda, Pacific, raw	2.6	75.4	0.0
E: shellfish				
16E-01	fish/shellfish: abalone, mixed species, raw	0.76	74.56	*
16E-02	fish/shellfish: clam, mixed species, raw	0.97	81.82	*
16E-04	fish/shellfish: mussel, blue, raw	2.24	80.58	*
16E-03	fish/shellfish: oyster, eastern, raw	2.47	85.14	*
16E-03	fish/shellfish: oyster, Pacific, raw	2.3	82.06	*
16E-05	fish/shellfish: scallop, mixed species, raw	0.76	78.57	*
16E-99	fish/shellfish: whelk, unspecified, raw	0.4	66	*
J: crustaceans				
16J-01	fish/shellfish: crab, Alaska king, raw	0.6	79.57	0.0
16J-01	fish/shellfish: crab, blue, raw	1.08	79.02	*
16J-01	fish/shellfish: crab, dungeness, raw	0.97	79.18	*
16J-01	fish/shellfish: crab, queen, raw	1.18	80.58	0.0
16J-02	fish/shellfish: crayfish, mixed species, raw	1.06	80.79	0.0
16J-04	fish/shellfish: lobster, northern, raw	0.9	76.76	*
16J-05	fish/shellfish: shrimp, mixed species, raw	1.73	75.86	*
16J-03	fish/shellfish: spiny lobster, mixed species, raw	1.51	74.07	*
M: other aquatic animals				
16M-07	fish/shellfish: cuttlefish, mixed species, raw	0.7	80.56	*
16M-09	fish/shellfish: octopus, common, raw	1.04	80.25	*
16M-03	fish/shellfish: squid, mixed species, raw	1.38	78.55	*
16M-01	frog legs: raw	0.3	81.9	0.0
16M-05	seafood: terrapin, (diamond back), raw	3.5	77	0.0
16M-05	seafood: turtle, green, raw	0.5	78.5	0.0
16M-06	seafood: whale meat, raw	7.5	70.9	*
R: engineered seafood				
16R-01	fish/shellfish: surimi	0.9	76.34	*
Y: fishery products				
16Y-04	fish/shellfish: roe, mixed species, raw	6.42	67.73	*

17: MEAT AND POULTRY

A: red meat products				
17A-01	frankfurters: raw, beef	28.54	54.71	*

Product Code	Commodity	% Fat	% Water	% Sugars
17A-99	frankfurters: raw, beef & pork	29.15	53.87	2
17A-99	goat: raw	2.31	75.84	*
B: poultry/poultry products				
17B-99	frankfurter: chicken	19.48	57.53	*
17B-06	frankfurter: turkey	17.7	62.99	*
20: FRUITS				
A: berries				
20A-01	blackberries: raw	0.39	85.64	7.9
20A-02	blueberries: raw	0.38	84.61	7.3
20A-04	cranberries: raw	0.2	86.54	*
20A-05	currants: European black, raw	0.41	81.96	8
20A-05	currants: red and white, raw	0.2	83.95	8
20A-07	elderberries: raw	0.5	79.8	*
20A-08	gooseberries: raw	0.58	87.87	*
20A-09	grapes: American type (slip skin), raw	0.35	81.3	16.4
20A-09	grapes: European type (adherent skin), raw	0.58	80.56	18.1
20A-99	groundcherries: (cape-gooseberries or poha), raw	0.7	85.4	*
20A-99	mulberries: raw	0.39	87.68	*
20A-99	oheloberries: raw	0.22	92.3	*
20A-10	raisins: golden seedless	0.46	14.97	*
20A-10	raisins: seeded	0.54	16.57	*
20A-10	raisins: seedless	0.46	15.42	61.7
20A-13	raspberries: raw	0.55	86.57	*
20A-14	strawberries: raw	0.37	91.57	5.7
D: berry juice				
20D-09	grape juice: canned or bottled, unsweetened	0.08	84.12	14.2
G: citrus fruit				
20G-02	grapefruit: raw, pink/red/white, all areas	0.1	90.89	6.2
20G-03	kumquats: raw	0.1	81.7	*
20G-04	lemon peel: raw	0.3	81.6	*
20G-04	lemons: raw, w/peel	0.3	87.4	*
20G-05	limes: raw	0.2	88.26	0.4
20G-06	oranges: raw, all commercial varieties	0.12	86.75	8.9
20G-06	oranges: raw, California, navels	0.09	86.81	*
20G-06	oranges: raw, California, valencias	0.3	86.34	*
20G-06	oranges: raw, Florida	0.21	87.14	8.9
20G-07	pummelo: raw	0.04	89.1	*
20G-09	tangerines: (mandarin orange), raw	0.19	87.6	*
K: citrus fruit juices				
20K-02	grapefruit juice: pink/red/white, all varieties, raw	0.1	90	7.5
20K-04	lemon juice: canned or bottled	0.29	92.46	2.4
20K-04	lemon juice: raw	0	90.73	2.4

Product Code	Commodity	% Fat	% Water	% Sugars
20K-05	lime juice: canned or bottled, unsweetened	0.23	92.52	*
20K-05	lime juice: raw	0.1	90.21	*
20K-06	orange juice: canned, unsweetened	0.14	89.01	9.8
20K-06	orange juice: raw	0.2	88.3	10.2
N: core fruit				
20N-01	apples: raw, w/skin	0.36	83.93	11.5
20N-02	crabapples: raw	0.3	78.94	*
20N-99	mammy-apple: (mamey), raw	0.5	86.2	*
20N-03	pears: raw	0.4	83.81	10.5
20N-05	pricklypears: raw	0.51	87.55	*
20N-04	quinces: raw	0.1	83.8	*
20N-99	rose-apples: raw	0.3	93	*
Q: core fruit, dried/paste				
20Q-01	apples: dehydrated (low moisture), sulfured, uncooked	0.58	3	*
20Q-01	apples: dried, sulfured, uncooked	0.32	31.76	*
20Q-01	applesauce: canned, sweetened, w/salt	0.18	79.58	16.5
S: core fruit juice				
20S-01	apple juice: canned or bottled, unsweetened, w/added asc acid	0.11	87.93	10.9

21: FRUIT PRODUCTS

G: pit fruit				
21G-01	apricots: raw	0.39	86.35	9.3
21G-02	avocados: raw, California	17.33	72.56	0.9
21G-02	avocados: raw, Florida	8.87	79.73	*
21G-03	cherries: sour, red, raw	0.3	86.13	8.1
21G-03	cherries: sweet, raw	0.96	80.76	14.6
21G-05	dates: domestic, natural and dry	0.45	22.5	64.2
21G-99	java-plum: (jambolan), raw	0.23	83.13	*
21G-16	jujube: raw	0.2	77.86	*
21G-08	loquats: raw	0.2	86.73	*
21G-07	nectarines: raw	0.46	86.28	8.5
21G-13	peaches: raw	0.09	87.66	8.7
21G-12	pitanga: (surinam-cherry), raw	0.4	90.81	*
21G-14	plums: raw	0.62	85.2	7.5
21G-99	sapodilla: raw	1.1	78	*
H: pit fruit dried/paste				
21H-01	apricots: dehydrated (low-moisture), sulfured, uncooked	0.62	7.5	*
21H-01	apricots: dried, sulfured, uncooked	0.46	31.09	38.9
K: pit fruit juice				
21K-01	apricot nectar: canned, w/added asc acid	0.09	84.87	13.5

Product Code	Commodity	% Fat	% Water	% Sugars
S: sub/tropical fruit				
21S-99	balsam-pear: leafy tips, raw	0.69	89.25	0.8
21S-99	balsam-pear: pods, raw	0.17	94.03	0.8
21S-02	bananas: raw	0.48	74.26	18.4
21S-02	bananas: red, raw	0.2	74.4	*
21S-20	carambola: (starfruit), raw	0.35	90.92	7.1
21S-99	cherimoya: raw	0.4	73.5	*
21S-99	custard-apple: (bullock's-heart), raw	0.6	71.5	*
21S-03	figs: raw	0.3	79.11	6.9
21S-04	guavas: common, raw	0.6	86.1	6
21S-04	guavas: strawberry, raw	0.6	80.66	*
21S-10	jackfruit: raw	0.3	73.23	18.4
21S-11	kiwifruit: (Chinese gooseberries), fresh, raw	0.44	83.05	8.9
21S-19	litchis: raw	0.44	81.76	*
21S-18	longans: raw	0.1	82.75	*
21S-05	mangoes: raw	0.27	81.71	14.8
21S-06	papayas: raw	0.14	88.83	5.9
21S-12	passion-fruit: (granadilla), purple, raw	0.7	72.93	11.2
21S-07	pineapple: raw	0.43	86.5	11.9
21S-08	plantain: raw	0.37	65.28	*
21S-16	sapotes: (marmalade plum), raw	0.6	62.43	*
21S-09	seeds: breadfruit seeds, boiled	2.3	59.3	*
21S-09	seeds: breadfruit seeds, raw	5.59	56.27	*
21S-14	tamarinds: raw	0.6	31.4	*
T: sub/tropical fruit dried/paste				
21T-19	litchis: dried	1.2	22.3	*
21T-18	longans: dried	0.4	17.6	*
V: sub/tropical fruit juice/milk,creme/nect				
21V-01	acerola juice: raw	0.3	94.3	*

22: VINE FRUITS (MELONS) AND PRODUCTS

A: vine fruit				
22A-01	melons: cantaloupe, raw	0.28	89.78	8.1
22A-02	melons: casaba, raw	0.1	92	*
22A-03	melons: honeydew, raw	0.1	89.66	*
22A-04	watermelon: raw	0.43	91.51	9
G: other fruit products				
22G-02	persimmons: Japanese, raw	0.19	80.32	*
22G-02	persimmons: native, raw	0.4	64.4	*
22G-01	pomegranate: raw	0.3	80.97	8.9
22G-99	roselle: raw	0.64	86.58	*
22G-04	soursop: raw	0.3	81.16	*
22G-05	sugar-apples: (sweetsop), raw	0.29	73.23	*

Product Code	Commodity	% Fat	% Water	% Sugars
23: NUTS, EDIBLE SEEDS, AND PRODUCTS				
A: nut in shell				
23A-04	nuts: chestnuts, European, raw, unpeeled	2.26	48.65	10.6
B: nut shelled				
23B-99	nuts: acorns, raw	23.86	27.9	*
23B-04	nuts: chestnuts, Chinese, raw	1.11	43.95	*
23B-04	nuts: chestnuts, European, raw, peeled	1.25	52	11.3
23B-04	nuts: chestnuts, Japanese, raw	0.53	61.41	*
23B-05	nuts: coconut meat, raw	33.49	46.99	3.5
23B-15	nuts: ginkgo nuts, raw	1.68	55.2	*
23B-07	peanuts: all types, raw	49.24	6.5	4.3
23B-07	peanuts: Spanish, raw	49.6	6.39	*
23B-07	peanuts: Valencia, raw	47.58	4.26	*
23B-07	peanuts: Virginia, raw	48.75	6.91	4.3
C: nut butter				
23C-07	nuts: peanut butter, w/salt added	49.98	1.42	*
23C-07	peanut butter: chunk style, w/salt	49.94	1.13	7.8
23C-07	peanut butter: chunk style, wo/salt	49.94	1.13	*
23C-07	peanut butter: smooth style, w/salt	49.98	1.42	7.8
23C-07	peanut butter: smooth style, wo/salt	49.98	1.42	7.8
K: edible seed				
23K-08	millet, raw	4.22	8.67	1.2
23K-09	quinoa	5.8	9.3	*
23K-06	seeds: lotus seeds, raw	0.53	77	*
23K-03	soybeans: green, raw	6.8	67.5	*
24: BEANS, PEAS, CORN, AND FRUITING VEGETABLES				
A: bean/pea/corn				
24A-01	alfalfa seeds: sprouted, raw	0.69	91.14	0.2
24A-99	beans: adzuki, mature seeds, raw	0.53	13.44	*
24A-99	beans: black turtle soup, mature seeds, raw	0.9	11	*
24A-16	beans: black, mature seeds, raw	1.42	11.02	*
24A-04	beans: cranberry (Roman), mature seeds, canned	0.28	77.56	*
24A-99	beans: French, mature seeds, raw	2.02	10.77	*
24A-10	beans: great northern, mature seeds, raw	1.14	10.7	*
24A-99	beans: hyacinth, mature seeds, raw	1.69	9.38	*
24A-07	beans: kidney, all types, mature seeds, raw	0.83	11.75	*
24A-07	beans: kidney, California red, mature seeds, raw	0.25	11.75	*
24A-07	beans: kidney, red, mature seeds, raw	1.06	11.75	*
24A-07	beans: kidney, royal red, mature seeds, raw	0.45	11.9	*
24A-08	beans: lima, large, mature seeds, raw	0.69	10.17	8.5
24A-09	beans: mung, mature seeds, raw	1.15	9.05	6.6
24A-01	beans: mung, mature seeds, sprouted, raw	0.18	90.4	1.7
24A-99	beans: mungo, mature seeds, raw	1.83	8.58	*

Product Code	Commodity	% Fat	% Water	% Sugars
24A-10	beans: navy, mature seeds, raw	1.28	12.36	*
24A-99	beans: pink, mature seeds, raw	1.13	10.06	*
24A-11	beans: pinto, mature seeds, raw	1.13	10.95	*
24A-99	beans: small white, mature seeds, raw	1.18	11.71	*
24A-14	beans: snap, green var, raw	0.12	90.27	2.6
24A-15	beans: snap, yellow var, raw	0.12	90.27	*
24A-10	beans: white, mature seeds, raw	0.85	11.32	*
24A-99	beans: winged, mature seeds, raw	16.32	8.34	7
24A-14	beans: yardlong, mature seeds, raw	1.31	8.43	*
24A-99	beans: yellow, mature seeds, raw	2.6	11.1	*
24A-05	broadbeans (fava beans): mature seeds, raw	1.53	10.98	5.7
24A-06	chickpeas (garbanzo beans, bengal gram): mature seeds, raw	6.04	11.53	3.8
24A-60	corn, white	4.74	10.37	*
24A-60	corn, yellow	4.74	10.37	*
24A-60	corn: sweet, white, raw	1.18	75.96	*
24A-60	corn: sweet, yellow, raw	1.18	75.96	5.4
24A-99	cowpeas: catjang, mature seeds, raw	2.07	11.05	3.0
24A-99	hyacinth-beans: immature seeds, raw	0.2	87.87	*
24A-70	lentils: mature seeds, raw	0.96	11.19	2.5
24A-99	lupins: mature seeds, raw	9.74	10.44	*
24A-99	mothbeans: mature seeds, raw	1.61	9.68	*
24A-99	natto	11	55.02	*
24A-51	peas: edible-podded, raw	0.2	88.89	4
24A-51	peas: green, raw	0.4	78.86	4.5
24A-99	peas: split, mature seeds, raw	1.16	11.27	*
24A-17	pigeon peas (red gram): mature seeds, raw	1.49	10.59	2.8
24A-13	soybeans: mature seeds, raw	19.94	8.54	6.6
24A-99	tempeh	7.68	54.95	1.3
24A-13	tofu: raw, firm	8.72	69.83	0.4
24A-13	tofu: raw, regular	4.78	84.55	0.4
24A-99	winged bean: leaves, raw	1.1	76.85	*
24A-99	winged bean: tuber, raw	0.9	57.4	*
24A-14	yardlong bean: raw	0.4	87.85	*
F: fruit (vegetable)				
24F-99	chayote: fruit, raw (pear-shaped veg, squash family)	0.3	93	*
24F-20	cucumber: not pared, raw	0.13	96.05	2.3
24F-01	eggplant: raw	0.1	91.93	3.4
24F-09	gourd: dishcloth (towelgourd), raw	0.2	93.85	*
24F-09	gourd: white-flowered (calabash), raw	0.02	95.54	*
24F-02	okra: raw	0.1	89.58	2.4
24F-08	peppers: hot chili, green, raw	0.2	87.74	*
24F-08	peppers: hot chili, red, raw	0.2	87.74	*
24F-07	peppers: sweet, green, raw	0.19	92.19	2.5
24F-07	peppers: sweet, red, raw	0.19	92.19	*
24F-05	pumpkin: raw	0.1	91.6	4.4
24F-06	squash: summer, all varieties, raw	0.21	93.68	2.2

Product Code	Commodity	% Fat	% Water	% Sugars
24F-06	squash: summer, crookneck and straightneck, raw	0.24	94.2	2.2
24F-06	squash: summer, scallop, raw	0.2	94.18	2.2
24F-06	squash: summer, zucchini, incl skin, raw	0.14	95.28	2.2
24F-06	squash: winter, acorn, raw	0.1	87.78	2.2
24F-06	squash: winter, all varieties, raw	0.23	88.72	2.2
24F-06	squash: winter, butternut, raw	0.1	86.41	2.2
24F-06	squash: winter, hubbard, raw	0.5	88	2.2
24F-06	squash: winter, spaghetti, raw	0.57	91.6	2.2
24F-50	tomatoes: green, raw	0.2	93	2.7
24F-50	tomatoes: red, ripe, raw, yr round average	0.33	93.76	3.0
24F-09	waxgourd: (Chinese preserving melon), raw	0.2	96.1	1
T: leaf/stem vegetable				
24T-99	amaranth	6.51	9.84	1.9
24T-01	artichokes: (globe or French), raw	0.15	84.94	2.2
24T-02	asparagus: raw	0.22	92.25	2.1
24T-03	bamboo shoots: raw	0.3	91	*
24T-04	beet greens: raw	0.06	92.15	*
24T-99	borage: raw	0.7	93	0.9
24T-05	broccoli: raw	0.35	90.69	1.6
24T-07	brussels sprouts: raw	0.3	86	2.2
24T-99	butterbur: (fuki), raw	0.04	94.5	*
24T-33	cabbage: Chinese (pak-choi), raw	0.2	95.32	1
24T-12	cabbage: Chinese (pe-tsai), raw	0.2	94.39	1.3
24T-08	cabbage: common (Danish, domestic, and pointed types), raw	0.18	92.52	2.7
24T-08	cabbage: red, raw	0.26	91.55	5.4
24T-08	cabbage: savoy, raw	0.1	91	2.9
24T-99	cardoon: raw	0.1	94	1.7
24T-10	cauliflower: raw	0.18	92.26	2.2
24T-44	celeriac: raw	0.3	88	2
24T-11	celery: raw	0.14	94.64	1
24T-99	celtuce: raw	0.3	94.5	1.7
24T-26	chard: Swiss, raw	0.2	92.66	1.1
24T-34	chicory: greens, raw	0.3	92	0.9
24T-34	chicory: witloof, raw	0.1	95.1	*
24T-13	collards: raw	0.22	90.55	*
24T-35	cornsalad: raw	0.4	92.8	*
24T-99	cowpeas: leafy tips, raw	0.25	89.78	*
24T-99	cress: garden, raw	0.7	89.4	*
24T-14	dandelion greens: raw	0.7	85.6	2.4
24T-99	dock: raw	0.7	93	*
24T-30	endive: raw	0.2	93.79	1.2
24T-99	horseradish-tree: leafy tips, raw	1.4	78.66	*
24T-99	jute, potherb: raw	0.25	87.72	*
24T-18	kale, Scotch: raw	0.6	87	2.2
24T-18	kale: raw	0.7	84.46	2.2
24T-99	lambsquarters: raw	0.8	84.3	*

Product Code	Commodity	% Fat	% Water	% Sugars
24T-31	lettuce: butterhead (includes Boston and bibb types), raw	0.22	95.58	*
24T-32	lettuce: cos or romaine, raw	0.2	94.91	2
24T-31	lettuce: iceberg (includes crisphead types), raw	0.19	95.89	1.8
24T-32	lettuce: looseleaf, raw	0.3	94	*
24T-20	mustard greens: raw	0.2	90.8	0.8
24T-99	mustard spinach: (tendergreen), raw	0.3	92.2	*
24T-25	New Zealand spinach: raw	0.2	94	*
24T-21	parsley: raw	0.3	88.31	1.1
24T-43	pokeberry shoots: (poke), raw	0.4	91.6	*
24T-99	purslane: raw	0.1	93.92	*
24T-24	rhubarb: raw	0.2	93.61	0.9
24T-29	seaweed: agar, raw	0.03	91.32	*
24T-29	seaweed: dulse, raw	3.2	16.6	*
24T-29	seaweed: irishmoss, raw	0.16	81.34	*
24T-29	seaweed: kelp, raw	0.56	81.58	*
24T-29	seaweed: laver, raw	0.28	85.03	*
24T-29	seaweed: spirulina, raw	0.39	90.67	*
24T-29	seaweed: wakame, raw	0.64	79.99	*
24T-99	sesbania flower: raw	0.04	91.58	*
24T-21	spices: parsley, dried	4.431	9.02	*
24T-25	spinach: raw	0.35	91.58	0.4
24T-42	swamp cabbage: (skunk cabbage), raw	0.2	92.47	*
24T-27	turnip greens: raw	0.3	91.07	1
24T-99	vinespinach: (basella), raw	0.3	93.1	*
24T-28	watercress: raw	0.1	95.11	0.4
25: VEGETABLES AND PRODUCTS				
J: root/tuber vegetable				
25J-28	arrowhead: raw	0.29	72.48	*
25J-08	beets: raw	0.14	87.32	5.9
25J-22	burdock root: raw	0.15	80.09	*
25J-01	carrots: raw	0.19	87.79	6.6
25J-16	cassava: raw	0.39	68.51	1.2
25J-20	chicory: roots, raw	0.2	80	2.4
25J-21	garlic: raw	0.5	58.58	1
25J-24	ginger root: raw	0.73	81.67	*
25J-02	horseradish-tree: pods, raw	0.2	88.2	*
25J-02	horseradish: raw	0.3	74.6	1.8
25J-29	Jerusalem-artichokes: raw	0.01	78.01	2.5
25J-99	kohlrabi: (thickened bulb-like stems): raw	0.1	91	4.5
25J-03	leeks: (bulb and lower leaf-portion), raw	0.3	83	3.9
25J-18	lotus root: raw	0.1	79.1	*
25J-12	mountain yam: Hawaii, raw	0.1	81.44	*
25J-25	onions: raw	0.16	89.68	4.1
25J-04	onions: spring (includes tops and bulb), raw	0.19	89.83	3.2
25J-25	onions: Welsh, raw	0.4	90.5	*
25J-05	parsnips: raw	0.3	79.53	4.8

Product Code	Commodity	% Fat	% Water	% Sugars
25J-06	potatoes: raw, flesh	0.1	78.96	1.0
25J-06	potatoes: raw, skin	0.1	83.29	0.6
25J-26	radishes: oriental, raw	0.1	94.62	2.5
25J-07	radishes: raw	0.54	94.84	2.7
25J-07	radishes: white icicle, raw	0.1	95.37	2.5
25J-09	rutabagas: raw	0.2	89.66	5.6
25J-10	salsify: (vegetable oyster), raw	0.2	77	2.9
25J-11	shallots, raw	0.1	79.8	3.2
25J-12	sweet potato leaves: raw	0.3	87.96	*
25J-12	sweet potatoes: raw	0.3	72.84	5.0
25J-23	taro, Tahitian: raw	0.97	87.96	*
25J-23	taro: leaves, raw	0.74	85.66	*
25J-23	taro: raw	0.2	70.64	0.8
25J-23	taro: shoots, raw	0.09	95.82	*
25J-14	turnips: raw	0.1	91.87	3.8
25J-15	water chestnuts: Chinese, (matai), raw	0.1	73.46	4.8
25J-12	yam: raw	0.17	69.6	0.5
25J-17	yambean: raw	0.2	89.15	*
L: root/tuber vegetable dried/paste/spread/f				
25L-23	poi	0.14	71.64	*
P: fungi, mushrooms, truffles; whole (button				
25P-01	mushrooms: raw	0.42	91.81	1.8
25P-04	mushrooms: shiitake, dried	0.99	9.5	*
S: fungi, mushroom, truffle products, not el				
25S-99	jew's ear: (pepeao), raw	0.04	92.59	*

28: SPICES, FLAVORS, AND SALTS

A: whole spices				
28A-99	chervil: raw	0.9	80.7	*
28A-99	chives: raw	0.6	92	1
28A-15	coriander: raw	0.59	92.8	*
28A-18	fennel: leaves, raw	0.4	90	*
28A-03	spices: anise seed	15.901	9.535	*
28A-09	spices: caraway seed	14.593	9.875	*
28A-12	spices: celery seed	25.271	6.037	*
28A-15	spices: coriander seed	17.77	8.861	*
28A-16	spices: cumin seed	22.27	8.063	*
28A-17	spices: dill seed	14.535	7.701	*
28A-18	spices: fennel seed	14.868	8.813	*
28A-56	spices: fenugreek seed	6.408	8.843	*
28A-29	spices: mustard seed, yellow	28.759	6.858	*
28A-37	spices: poppy seed	44.704	6.782	*
28A-40	spices: saffron	5.852	11.898	*

Product Code	Commodity	% Fat	% Water	% Sugars
B: ground/cracked spices				
28B-43	seeds: sesame butter, tahini, from raw and stone ground kernels	48	3	*
28B-01	spices: allspice, ground	8.685	8.459	*
28B-04	spices: basil, ground	3.977	6.432	*
28B-05	spices: bay leaf, crumbled	8.362	5.436	*
28B-10	spices: cardamom, ground	6.699	8.282	*
28B-99	spices: chervil, dried	3.9	7.2	*
28B-08	spices: chili powder	16.76	7.79	*
28B-13	spices: cinnamon, ground	3.185	9.52	*
28B-14	spices: cloves, ground	20.066	6.857	*
28B-15	spices: coriander leaf, dried	4.778	7.3	*
28B-17	spices: dill weed, dried	4.36	7.3	*
28B-11	spices: garlic powder	0.759	6.446	*
28B-19	spices: ginger, ground	5.949	9.377	*
28B-24	spices: mace, ground	32.382	8.172	*
28B-27	spices: marjoram, dried	7.036	7.641	*
28B-30	spices: nutmeg, ground	36.307	6.228	*
28B-31	spices: onion powder	1.052	5.005	*
28B-27	spices: oregano, ground	10.25	7.164	*
28B-33	spices: paprika	12.953	9.536	*
28B-54	spices: pepper, black	3.26	10.508	*
28B-08	spices: pepper, red or cayenne	15.668	8.047	*
28B-55	spices: pepper, white	2.12	11.42	*
28B-53	spices: rosemary, dried	15.22	9.306	*
28B-41	spices: sage, ground	12.745	7.955	*
28B-42	spices: savory, ground	5.907	9.003	*
28B-47	spices: tarragon, ground	7.242	7.739	*
28B-48	spices: thyme, ground	7.425	7.79	*
28B-49	spices: turmeric, ground	9.876	11.356	*
F: ground/cracked mix spice/season w/o salt				
28F-04	spices: curry powder	13.81	9.52	*
36: SWEETENERS (NUTRITIVE)				
C: honey				
36C-02	honey: strained or extracted	0	17.2	81.9
38: SOUPS				
A: soup				
38A-33	miso	6.07	41.45	*
40: BABY FOODS				
A: baked goods (baby)				
40A-01	babyfood: cookies, arrowroot	14.3	5.6	23
40A-02	babyfood: teething biscuits	4.2	6.4	24
40A-02	zwieback	9.7	4.5	12.9

Product Code	Commodity	% Fat	% Water	% Sugars
B: cereal (baby)				
40B-01	babyfood: cereal, barley, dry	3.4	6.8	9.79
40B-30	babyfood: cereal, egg yolks and bacon, str	5.2	84.9	*
40B-03	babyfood: cereal, hi-prot, dry	5.9	6.1	3.89
40B-10	babyfood: cereal, mixed, dry	4.4	6.7	7.75
40B-20	babyfood: cereal, mixed, w/applesauce & bananas, junior	0.4	79.6	11
40B-20	babyfood: cereal, mixed, w/applesauce & bananas, strained	0.5	80	11
40B-20	babyfood: cereal, mixed, w/bananas, dry	4.6	4.5	20.9
40B-04	babyfood: cereal, oatmeal, dry	7.8	6.2	9.79
40B-20	babyfood: cereal, oatmeal, w/applesauce & bananas, junior	0.7	81.8	10.4
40B-20	babyfood: cereal, oatmeal, w/applesauce & bananas, strained	0.7	82.2	10.4
40B-20	babyfood: cereal, oatmeal, w/bananas, dry	6	4.7	22.4
40B-05	babyfood: cereal, rice, dry	4.9	6.7	9.75
40B-20	babyfood: cereal, rice, w/applesauce & bananas, strained	0.4	81	8.91
40B-20	babyfood: cereal, rice, w/bananas, dry	4.2	4.7	19.4
40B-30	babyfood: cereal, w/egg yolks, junior	1.8	88.7	0.68
40B-30	babyfood: cereal, w/egg yolks, strained	1.8	88.8	0.68
D: vegetables (baby)				
40D-06	babyfood: vegetables, carrots, jnr	0.2	91	3.07
40D-06	babyfood: vegetables, carrots, str	0.1	92.3	3.07
40D-02	babyfood: vegetables, corn, creamed, junior	0.4	81.4	1.18
40D-02	babyfood: vegetables, corn, creamed, strained	0.4	83.6	1.18
40D-01	babyfood: vegetables, green beans, buttered, jnr	0.9	91.2	*
40D-01	babyfood: vegetables, green beans, buttered, str	0.8	90.8	*
40D-01	babyfood: vegetables, green beans, jnr	0.1	92.5	*
40D-05	babyfood: vegetables, mix veg, jnr	0.4	89.4	1.36
40D-05	babyfood: vegetables, mix veg, str	0.5	89.8	1.36
40D-01	babyfood: vegetables, peas, creamed, strained	1.9	86.5	1.52
40D-04	babyfood: vegetables, spinach, creamed, jnr	1.4	88.2	1.94
40D-04	babyfood: vegetables, spinach, creamed, str	1.3	89.6	1.94
40D-03	babyfood: vegetables, squash, jnr	0.2	92.8	2.18
40D-03	babyfood: vegetables, squash, str	0.2	92.7	2.18
E: fruit/juice/drink (baby)				
40E-20	babyfood: fruit, apple & blueberry, junior	0.2	82.8	8.9
40E-20	babyfood: fruit, apple & blueberry, strained	0.2	83.1	8.9
40E-20	babyfood: fruit, applesauce & apricots, jnr	0.2	86.9	10.6
40E-20	babyfood: fruit, applesauce & apricots, str	0.2	87.7	10.6
40E-20	babyfood: fruit, applesauce & pineapple, junior	0.1	89.1	11.8
40E-20	babyfood: fruit, applesauce & pineapple, strained	0.1	89.5	11.8
40E-20	babyfood: fruit, pears & pineapple, jnr	0.2	87.8	8.58
40E-20	babyfood: fruit, pears & pineapple, str	0.1	88.5	8.58
40E-10	babyfood: fruit, pears, jnr	0.1	87.8	10.6
40E-10	babyfood: fruit, pears, str	0.2	88.4	10.6
40E-03	babyfood: juice, apple	0.1	88	14
40E-30	babyfood: juice, apple & grape	0.2	88.1	13.3

Product Code	Commodity	% Fat	% Water	% Sugars
40E-30	babyfood: juice, apple & peach	0.1	89	11.4
40E-30	babyfood: juice, apple & prune	0.1	81.3	13.1
40E-30	babyfood: juice, mixed fruit	0.1	87.9	*
40E-02	babyfood: juice, orange	0.3	88.5	*
40E-30	babyfood: juice, orange & apple	0.2	88.9	*
40E-30	babyfood: juice, orange & apple & banana	0.1	87.6	12.3
40E-30	babyfood: juice, orange & pineapple	0.1	87.3	12
F: meat products/comb meat dinner (baby)				
40F-51	babyfood: dinner, beef noodle, jnr	1.9	87.8	*
40F-50	babyfood: dinner, macaroni & tomato & beef, jnr	1.1	86.7	1.44
40F-50	babyfood: dinner, macaroni & tomato & beef, str	1.1	87.3	1.44
40F-53	babyfood: dinner, spaghetti & tomato & meat, jnr	1.3	85.5	1.21
40F-53	babyfood: dinner, spaghetti & tomato & meat, toddler	1	81.6	*
40F-55	babyfood: dinner, veg & bacon, jnr	3.9	86.2	1.71
40F-55	babyfood: dinner, veg & bacon, str	3.3	85.9	1.71
40F-57	babyfood: dinner, veg & ham, jnr	1.7	88.4	1.26
40F-57	babyfood: dinner, veg & ham, str	1.7	89.2	1.26
40F-57	babyfood: dinner, veg & ham, toddler	3	83.6	1.26
40F-58	babyfood: dinner, veg & lamb, jnr	1.7	88.6	1.08
40F-58	babyfood: dinner, veg & lamb, str	2	88.6	1.08
40F-59	babyfood: dinner, veg & liver, jnr	0.6	88.9	1.03
40F-59	babyfood: dinner, veg & liver, str	0.4	90	1.03
40F-01	babyfood: hi-mt dinner, beef & all veg, str	4.2	85.4	0.71
40F-01	babyfood: hi-mt dinner, beef & veg, jnr	4.6	83.2	0.71
40F-01	babyfood: meat, beef with beef heart, strained	4.4	82.5	*
40F-01	babyfood: meat, beef, junior	4.9	79.9	*
40F-01	babyfood: meat, beef, strained	5.4	80.6	*
40F-99	babyfood: meat, ham, jnr	6.7	78.5	*
40F-99	babyfood: meat, ham, str	5.8	79.4	*
40F-02	babyfood: meat, lamb, junior	5.2	79.6	*
40F-02	babyfood: meat, lamb, strained	4.7	80.3	*
40F-03	babyfood: meat, liver, strained	3.8	79.3	*
40F-54	babyfood: meat, meat sticks, junior	14.6	69.5	*
40F-99	babyfood: meat, pork, strained	7.1	78.4	*
40F-04	babyfood: meat, veal, junior	5	79.8	*
40F-04	babyfood: meat, veal, strained	4.8	80.9	*
G: poultry product/comb poultry dinner (baby)				
40G-40	babyfood: dinner, veg & chicken, jnr	1.1	88.2	1.17
40G-40	babyfood: dinner, veg & chicken, str	1.1	90	1.17
40G-40	babyfood: hi-mt dinner, chicken & veg, jnr	5.5	82.7	0.53
40G-40	babyfood: hi-mt dinner, chicken & veg, str	3.6	83.7	*
40G-50	babyfood: hi-mt dinner, turkey & veg, jnr	5	82.5	0.71
40G-50	babyfood: hi-mt dinner, turkey & veg, str	4.8	83	*
40G-20	babyfood: meat, chicken sticks, junior	14.4	68.3	*
40G-01	babyfood: meat, chicken, jnr	9.6	76	*

Product Code	Commodity	% Fat	% Water	% Sugars
40G-01	babyfood: meat, chicken, str	7.9	77.5	*
40G-20	babyfood: meat, turkey sticks, junior	14.2	69.8	*
40G-02	babyfood: meat, turkey, junior	7.1	77.5	*
40G-02	babyfood: meat, turkey, strained	5.8	78.9	*
H: high meat dinner/cheese food (baby)				
40H-30	babyfood: dinner, macaroni & cheese, junior	2	86.5	1.2
40H-30	babyfood: dinner, macaroni & cheese, strained	2.1	87.1	1.2
40H-30	babyfood: hi-mt dinner, cottage cheese w/pineapple, strained	2.2	72	*
J: egg products (baby)				
40J-01	babyfood: egg yolks, strained	17.3	70.6	*
K: pudding/custard (baby)				
40K-99	babyfood: dessert, caramel pudding, junior	0.9	80.4	*
40K-99	babyfood: dessert, cherry vanilla pudding, junior	0.2	81	10.6
40K-09	babyfood: dessert, custard pudding, vanilla, jnr	2.3	79.4	*
40K-99	babyfood: fruit, apricot w/tapioca, jnr	0	82.1	12.6
40K-99	babyfood: fruit, apricot w/tapioca, str	0	83.1	12.6
40K-99	babyfood: fruit, bananas & pineapple with tapioca, jnr	0.1	81.1	6.67
40K-99	babyfood: fruit, bananas & pineapple with tapioca, str	0	81.7	6.67
40K-99	babyfood: fruit, bananas with tapioca, jnr	0.2	81.5	11.6
40K-99	babyfood: fruit, bananas with tapioca, strained	0.1	84	11.6
L: soups/soup mix (baby)				
40L-02	babyfood: dinner, chicken soup, strained	1.7	89.1	0.67
Y: baby food, not elsewhere classified				
40Y-99	babyfood: dessert, dutch apple, junior	1	82.1	12.3
40Y-99	babyfood: dessert, dutch apple, strained	0.9	82.2	12.3
40Y-99	babyfood: dessert, peach cobbler, junior	0	81.2	14.1
40Y-99	babyfood: dessert, peach cobbler, strained	0	81.8	14.1

202: BASIC ANALYTICAL TECHNIQUES

202 A: INTRODUCTION

Most residue analytical methods follow the same patterns and use the same techniques. This section provides step-by-step recommended operating procedures for certain commonly used analytical techniques that appear repeatedly in the methods described in Chapters 3 and 4 of this manual. These procedure descriptions are not aimed at experienced analysts but may be useful to the relatively inexperienced. Justifications for certain steps are provided to offer guidance to the analyst troubleshooting an operation that did not perform as expected.

The techniques described in this section will not be described in detail elsewhere in PAM I. Where these techniques are used in the methods of Chapters 3 and 4, only the information particular to the method will be prescribed. Where alternative techniques appear in this section, the one most appropriate to a particular method is described in Chapters 3 and 4; the analyst is responsible for validating its replacement with an alternative.

202 B: COLUMN CHROMATOGRAPHY

Column chromatography is used to "clean up" extracts, *i.e.*, to remove extraneous materials that were co-extracted from the commodity with the residues. Ideally, co-extractives such as fat and chlorophyll are more strongly retained by the column adsorbent than are the residues. If so, solvent can be passed through the column, dissolving and removing ("eluting") residues, while leaving co-extractives attached to the adsorbent. Sequential elution of the column with different mixtures of solvents may also be used to separate groups of residues from one another.

The ability of a particular column chromatographic system to remove co-extractives and separate residues is determined empirically during method development. Successful duplication of results by other analysts requires adherence to both recommended operating procedures and specific directions in the method. This section provides terminology, recommended operating procedures, and suggestions for dealing with common problems encountered in the use of column chromatography.

Terminology

adsorbent: a finely sieved solid material, usually of prescribed mesh size, to which dissolved substances will preferentially attach and thus be removed from solutions.

column: either an empty glass tube intended to hold an adsorbent or that tube filled with adsorbent. Residue analytical methods most often require columns with fritted glass discs at the bottom, to hold the adsorbent in place, and Teflon stopcocks to control the rate at which eluant passes through the adsorbent; column length and diameter vary. Columns without stopcocks are suitable only for drying extracts through anhydrous sodium sulfate. If a column has no fritted glass disc, a plug of glass wool is placed in the bottom to retain the adsorbent. Solvent reservoirs incorporated at the top of columns are an optional convenience.

eluant: solvent or mixture of solvents that is passed through the column to remove ("elute") adsorbed residues; also known as eluting solvent.

eluate: solvent or mixture of solvents that has passed through the column. The eluate is the cleaned up extract.

Recommended Operating Procedure

- If adsorbent is stored in oven, remove and allow to reach room temperature in desiccator before use.
- Rinse empty glass column with suitable solvent to remove contaminants.
- Depending on method, either weigh appropriate amount of adsorbent and pour it into empty glass column or pour enough adsorbent into column to reach prescribed height.
- Gently tap side of column to settle adsorbent. Most laboratories make use of empty cardboard tube, length of rubber hose, or some other homemade device for this purpose.
- If directions specify layer of anhydrous sodium sulfate or glass wool plug, add on top of adsorbent layer. Gently tap again.
- Open stopcock completely and pour prescribed volume of rinse solvent through adsorbent; tap again gently during rinsing. Close stopcock when solvent level is still slightly above adsorbent. Discard rinse solvent.
- Place receiving vessel prescribed by method under column tip. Open stopcock part way and immediately transfer sample extract to top of column. As solvent drips from bottom, rinse vessel from which extract was poured with several small volumes of same solvent in which extract is dissolved; add rinses to column.
- As last rinse approaches top of adsorbent layer, add prescribed volume of eluant to column. Adjust stopcock to create prescribed flow rate of eluant through column. Allow elution to proceed; do not use pressure or vacuum to speed elution unless directed by method.
- If method requires more than one eluant, close stopcock when first eluant is still somewhat above top of adsorbent layer. Change receiving vessel, open stopcock part way, and immediately add prescribed amount of next eluant to top. Adjust flow rate as before, and repeat as needed.

Common Problems

Variation in Adsorptivity. Column chromatography is useful for cleanup because of the adsorbent's capacity to retain materials; this "adsorptivity" is dependent on both physical and chemical characteristics. Adsorptivity of a material may vary from one batch to another, with variations affecting both adequacy of cleanup and recovery of residues. To minimize problems caused by variations in adsorptivity, follow these rules:

- Always purchase exact material specified by method. Although relatively few materials are commonly used as adsorbents, many brands and manufacturing treatments are available. Methods are developed using specific adsorbents, and substitutes should not be used.

- Carefully follow any adsorbent handling and storage procedures specified to recreate conditions used during method development.

- If method requires heating adsorbent, always cool in desiccator before use to prevent absorption of moisture from air, which will change adsorptivity.

- If available, use calibration steps provided in method to measure, verify, and/or compensate for variations in adsorptivity.

Physical Disruption of Adsorbent Column. Because adsorption occurs while dissolved materials are in contact with the surface of the adsorbent, cleanup and separations may be adversely affected when physical disruption of the column occurs. Improper handling can cause "pockets" or "channels" to develop in the adsorbent column; if these exist, solutions will flow preferentially through those spaces rather than spread evenly throughout the adsorbent, thus decreasing solute exposure to the adsorbent surface area. Reduction of cleanup capacity and change in elution patterns will likely occur.

To minimize problems caused by channeling, follow the recommended operating procedure, especially:

- Cool adsorbent before use in column; solvents may boil in presence of hot adsorbent and leave pockets within column.

- Tap adsorbent to settle it properly.

- Do not let column go dry; always close stopcock when eluant level is still somewhat above top of adsorbent layer.

Contribution of Interferences. Any material used in residue analysis is a potential source of interference during the determinative steps of the analysis. Routine performance of "reagent blanks," *i.e.*, complete method with no sample present, will notify the analyst if adsorbents or other reagents used in the method are contributing contaminants that interfere. Once detected, the source of contamination must be eliminated. To minimize interferences from adsorbents:

- Always rinse adsorbent as directed and discard rinsings.

- Follow any method directions concerning prewashing adsorbent.

- Store adsorbent in manner that prevents contamination; *e.g.*, if stored without stopper, at least keep vessel covered with loose layer of aluminum foil.

202 C: SOLVENT EVAPORATION

Essentially all residue analytical methods require removal of solvent at some point in order to increase the concentration of the analyte(s) in solution. Several different concentration/evaporation techniques are available, each with advantages and disadvantages. The best technique in a particular situation depends on the physical and chemical characteristics of the analyte and the solvent that must be evaporated.

Each method in Chapters 3 and 4 directs use of a specific concentration/evaporation technique. The method developer chose the particular technique for its applicability to the analyte and solvent involved, so attempts by the user to substitute a different technique will be successful only if the substitute is also applicable. This section provides information on applicability of several techniques, directions for correct operation, and cautions about common problems.

One evaporation technique not described in this section is evaporation in an open vessel, with or without gas flow impinging upon the solvent. Evaporating solvent in this way is not recommended for quantitative residue analysis, because loss of residues is likely [1], opportunities for contamination are increased, and safety and hazardous waste concerns are increased compared to other techniques.

Kuderna-Danish Concentrators

Use of Kuderna-Danish (K-D) concentrators for evaporation of solvents was established in FDA pesticide residue analyses during the 1960s when studies proved its value in reducing large volumes of solvent quickly and without loss of analytes [2]. Most methods in Chapter 3 specify K-D evaporation, because this technique was the most reliable at the time the methods underwent interlaboratory validation.

Application. Evaporation in K-D is most suitable for solvents with relatively low boiling points, such as ethyl ether (b.p. 34.6° C), methylene chloride (40.5°), and acetone (56.5°), or for solvent mixtures that form low boiling azeotropes. Analytes must be able to withstand steam bath temperatures for concentration by K-D.

Equipment. Basic K-D equipment is available from most major suppliers of laboratory equipment; certain sizes may be available only from specialty glassware suppliers.

Complete K-D apparatus consists of three pieces:

1) receiving flask, 10-50 mL volume, ground glass joint. Flasks may be straight sided tubes, most useful for concentration of extracts that will be transferred to other containers, or volumetric or graduated flasks, useful for concentration of final, cleaned up extract. No transfer is necessary if volume of final solution can be reliably measured in receiving flask. Ground glass joint at top of receiving flask must match that at bottom of concentrator.

2) concentrator, available in 125, 250, and 500 mL sizes, with ground glass joints at bottom and top. Concentrator holds most of the solvent during evaporation and is empty at end of procedure.

3) Snyder column, specially designed 30 cm long condensation column containing three balls; permits escape of solvent in gas phase without loss of higher boiling analytes. Micro-Snyder columns, with two balls, and micro-Vigreaux columns, with no balls, are also available for use in evaporating solvent in the receiving flask only, without use of concentrator.

Boiling chips facilitate solvent evaporation by providing a surface area for bubbles to form as boiling starts; use of 20-30 mesh carborundum chips minimizes volume displaced by boiling chip.

Recommended Operating Procedure.

- Connect concentrator and receiving flask and secure with springs or other means of preventing separation.

- Collect solutions from extraction or cleanup steps in connected concentrator/receiving flask.

- Turn on steam in steam bath.

- Add boiling chip to receiving flask, place Snyder column on top of concentrator, and gently lower receiving flask into opening in steam bath. Until boiling is well established, hold in place and tap side of Snyder column to facilitate pressure release. (Excess pressure buildup will result in loss of sample if sudden release occurs.)

- Support concentrator on steam bath. Adjust steam as needed to ensure appropriate evaporation rate. Once started, boiling should cause balls in Snyder column to rattle vigorously as escaping gases move past them. Do not allow boiling to be so vigorous that Snyder column is flooded with liquid.

- When balls in Snyder column stop rattling, remove K-D from steam bath and allow to cool, with Snyder column in place, until all fluid has drained back into receiving flask. Volume can be reduced to ≥5 mL in this way.

- Follow method directions for reconcentration in presence of additional solvent volume or different solvent, as sometimes required for complete removal of particular solvent.

- To reduce volume to <5 mL, remove receiving flask from concentrator and add fresh boiling chip. Connect micro-Snyder column directly to receiving flask. (To avoid flooding two-ball micro-Snyder column during evaporation, use micro-Vigreaux or other micro reflux column without balls for solvents with boiling point >65° C.)

- Hold receiving flask with spring test tube holder, and place tip into steam. Position tip of receiving flask carefully to avoid "bumping" of solvent as it boils. Allow to boil until volume is slightly less than desired.

- Allow apparatus to cool and fluid to drain into receiving flask before removing column. Minimum attainable volume is about 0.2-0.4 mL.

Rotary Evaporator

Vacuum and heat combine to reduce the quantity of solvent in a rotary evaporator. Solution in a round-bottom (r-b) flask is simultaneously rotated and heated by a water bath, while a vacuum pulled from the end of a condenser increases the rate of evaporation. Solvent evaporated from the solution is collected in a second flask attached to condenser.

Application. Evaporation of solvent using rotary evaporation is recommended for heat-labile residues, because temperature of the water bath used is much lower than that of a steam bath. Vacuum withdrawal of vapors, combined with water bath temperature of about 30° C, adequately removes solvents such as methylene chloride (b.p. 40.5° C) without damaging analytes. Evaporation in a rotary evaporator is more rapid than in a K-D, but is limited to only one solution at a time.

Equipment.

1) r-b flasks of appropriate volume. Flask that contains solution being evaporated has ground glass joint; flask that collects condensate has ball-and-socket joint.

2) rotary evaporator, including condenser and variable speed motor that rotates shaft to which r-b flask is attached. Use of glass trap connected to shaft between motor and r-b flask is recommended to protect motor. Electronically controlled motor for constant torque is preferred to maintain constant rotation throughout evaporation. Variable transformer for controlling speed is recommended only for evaporator with AC/DC motor; use of variable transformer on evaporator with AC motor will result in burning out evaporator motor.

3) constant temperature water bath

4) vacuum pump

5) needle valve to control vacuum, positioned between condenser and pump. Controlling vacuum with stopcock-type valve on condenser is also possible but is less precise than needle valve and requires more analyst attention. Use of vacuum gauge (preferred) or manometer is also recommended.

6) (optional) additional traps between condenser and vacuum pump, to protect pump from effects of corrosive vapors. Refrigerated condensation traps and/or chemical traps that collect vapors on disposable cartridges are available.

Recommended Operating Procedure.

- Circulate cold water through condenser.
- Heat water in water bath to temperature specified in method.
- Attach r-b flask for collecting condensate to condenser with clip supplied with rotary evaporator.

- When temperatures have equilibrated, attach r-b flask containing solution to rotary evaporator shaft (or to trap on shaft) and lower r-b flask into water bath. (Directions in some methods specify placing flask into water bath *after* applying vacuum.)

- Turn on motor and adjust rotation to about 70 rpm.

- Turn needle valve to disconnect vacuum pump from apparatus, turn on pump, then apply vacuum to system gradually by adjusting valve to minimize frothing or rapid boiling of solution.

- When solution is evaporated to dryness, reverse these steps to shut down system.

Rotary Evaporator with Circulating Chilled Liquid

Rotary evaporation can be made applicable to higher boiling solvents by addition of a refrigeration unit and pump to chill and circulate coolant through condenser and through bath for collecting condensate.

Application. Rotary evaporation with circulating chilled liquid is specified when solvent with higher boiling points, such as methanol (b.p. 64.7° C), acetonitrile (81.6°), and toluene (110.6°), must be removed from heat-labile analytes. Evaporation is facilitated by simultaneous use of warm water bath, vacuum, and condensation and collection of evaporated solvent at the low temperature provided by circulating refrigerated coolant.

Equipment. Figure 401-a (Section 401) illustrates arrangement of the following basic equipment.

1) r-b flasks, as above

2) rotary evaporator, as above. Condenser should be insulated (*e.g.*, with Styrofoam rings) to maintain low temperature for efficient condensation.

3) constant temperature water bath

4) vacuum pump, with needle valve control and vacuum gauge, as above

3) system capable of chilling coolant such as ethylene glycol (antifreeze) and circulating it through coil of insulated condenser; coolant also circulates through bath in which receiving flask is placed. Several commercial units are available.

5) optional additional traps, as above

Recommended Operating Procedure.

- Ensure that coolant level meets manufacturer's recommendation, then turn on refrigeration and circulating unit; follow manufacturer's directions for equilibration time needed to reach temperature specified in method.

- Follow directions specified for rotary evaporator, above, starting with "Heat water in water bath to temperature specified in method."

Turbo-Vap

This microprocessor-controlled apparatus from Zymark Corp. permits automated evaporation of small volumes of solvent using a patented gas vortex shearing action and mild thermal conditions. Solutions in 200 mL tubes are held in a temperature-controlled water bath while a flow of nitrogen creates a helical flow vortex within the liquid. An exhaust fan moves solvent vapors into a hose for delivery to any suitable hood or other vent. The system can be programmed to shut off when liquid level reaches specified point or after a specified time; this permits solvent evaporation during unattended operation. Up to six samples can be evaporated simultaneously.

No recommended operating procedure for Turbo-Vap is provided in this section, because it is not yet specified by any PAM I method; experiments with the apparatus are included in current method development projects.

Common Problems

Loss of Analyte. Evaporation steps are potential sources of analyte loss. Predictably, losses increase with analyte volatility and with decreasing final solution volume. Any evaporation of solution to dryness may cause analyte loss; even the presence of co-extractives may not prevent losses of the most volatile residues. Studies using open vessel evaporation showed no correlation between amount of co-extractives and losses that occurred during evaporation, and evidence suggested that the nature of co-extractives is more important than the amount [1]. More recent studies showed higher losses of α-BHC, a volatile residue, when evaporating extracts to dryness twice in a Turbo-Vap than when concentrating (never <2 mL) in a K-D [3]. Purified extracts (free of plant extractives and fat) are evaporated to dryness only when absolutely necessary, *e.g.*, when all traces of a solvent like methylene chloride must be removed to prevent interference in a determinative step.

References

[1] Chiba, M., and Morley, H.V. (1968) *J. Assoc. Off. Anal. Chem.* **51**, 55-62

[2] Burke, J.A., *et al.* (1966) *J. Assoc. Off. Anal. Chem.* **49**, 999-1003

[3] Parfitt, C.H., Jr. (Nov. 1991) "Miniaturized Multiresidue Approach to Determine Pesticide Residues in Fresh Fruits and Vegetables," LIB 3616, FDA, Rockville, MD

203: EQUIPMENT AND PROCEDURES FOR COMMINUTING SAMPLES

Section 102 C describes regulatory requirements related to compositing and comminuting test samples so that the test portion removed for analysis can be considered representative of the original consignment. This section provides descriptions of equipment and procedures that FDA laboratories have found effective in comminuting specific commodities.

203 A: EQUIPMENT

Distinctions among various pieces of equipment often relate to the type of sample for which each is most effective. Some equipment is designed to work best with samples that are inherently liquid and thus easily mixed, while other devices provide the power necessary to cut dry products into small pieces for subsequent mixing and homogenization. The following categories of equipment are defined by their mode of operation and the commodity types for which they are most useful.

Blenders and Homogenizers

Devices labelled "blenders" or "homogenizers" usually include blades that are capable of high speed movement but are small relative to the total volume of the container. The container ("blender jar") is designed to propel the material being mixed into a vortex, so that it repeatedly comes into contact with the blades. Such devices are most effective with liquids or materials that liquefy readily when blended. Blenders and homogenizers are most often used in residue analysis for extraction of residues into a solvent that has been added to already chopped sample, but the same devices can also be used for homogenizing commodities that are primarily liquid.

Commercially available devices used for this purpose include Waring, Lourdes, Polytron, and Omni-Mixer models. At least one published study [1] showed that results obtained from using the first three of these were equivalent for practical purposes.

Choppers and Food Processors

Large capacity (20-40 qt) choppers, with blades designed to cut and mix simultaneously, are the traditional equipment used to comminute solid raw agricultural commodities, such as fruits and vegetables, into a homogenate from which test portions are taken. Depending on the water content of the commodity, the final chopped product will consist of a totally homogeneous slurry or a mixture of small pieces. A typical example of this equipment is the Hobart vertical cutter-mixer, originally designed for use in large volume food preparation industries.

Modern food processors, in commercial sizes, are also capable of chopping such commodities into sufficiently fine pieces to provide homogeneity. Because even commercial size food processors are smaller than the 20 qt Hobart cutter-mixers, processing of several batches, followed by thorough mixing, may be necessary.

Grinders

The presence of skin and cartilage in commodities such as raw meat and fish make homogenization difficult. Meat grinders, which force the product through openings in a plate, provide a better mechanism for homogenizing such products. Some choppers (above) can be equipped with grinder attachments so that the single device serves two purposes. Food processors (above) may also comminute these commodities to a suitably homogeneous state. In either case, freezing pieces of the product before grinding improves homogeneity of final mix.

Mills

Dry, hard commodities, such as grains, consist of small individual units, but need further comminuting to expose all parts of the product to the solvent used in the analytical method extraction step. Several types of mills have been found suitable for reducing commodities to particles of <20 mesh.

Mills such as the Wiley mill or Hammer mill grind the commodity with a shearing action created by metal blades rotating at high speed against metal cutting surfaces; a sieve ensures that only particles ground to less than a specified size are able to pass through and be collected. These devices have several drawbacks, including excessive heat buildup and difficult cleaning procedures. Most important, use of mills can cause commodity components to separate from one another and result in a nonhomogeneous final product, because soft, starchy components are reduced to smaller particles than are harder germ and coatings.

High power centrifugal mills are better than these traditional mills for grinding hard materials, including those with high oil content such as soybeans. Centrifugal mills operate by grinding the product with a multiple blade rotor while ground particles are moved through a sieve by centrifugal force. Most models offer a variety of blades and sieves, and the mill can be readily disassembled for cleaning. Capacity of these mills is limited, so processing of small batches and subsequent mixing is usually necessary, but operation of the mill is sufficiently fast that the effort is minimal.

203 B: PROCEDURES FOR SPECIFIC COMMODITIES

The following recommendations for comminuting or homogenizing specific commodities are based on FDA experiences. Once homogeneous material is prepared, a portion is removed and analyzed. If analysis is performed after the homogenate has been frozen, the homogenate must first be thawed completely and mixed thoroughly before a test portion can be taken for analysis; liquid that has separated during freezing and thawing must be re-incorporated.

Crabs and Crayfish

Crabs and crayfish that are marketed live must be sacrificed in order to remove inedible parts (specified in Section 102). Freeze, cook, or autoclave crabs to loosen meat from inedible parts; then prepare a homogenate of edible portion by grinding with a meat grinder, food processor, or chopper.

Eggs

Blend eggs in a Waring or other blender at low speed for ≥5 min or until composite is homogeneous. Low speed blending will minimize foaming or "whipping" of sample.

Fish

Preparation of homogeneous samples of fish depends on whether skin and/or bones are considered edible for the particular species and product. In all cases, preparation must meet the requirements of regulatory policies on what portion of the commodity to include in the composite (Section 102). Skin is removed from species whose skin is considered inedible (*e.g.*, catfish), as are other inedible portions, such as heads, tails, scales, fins, viscera, and inedible bones (Table 102-a). Water that results from thawing frozen fish should be discarded.

Grind the remaining composite three times in a meat grinder, food processor, or food cutter equipped with a grinder attachment [2]. If skin is included in the edible portion, freeze portions of suitable size before introduction into the grinder; this causes the skin to be more brittle and minimizes clogging of the grinder.

Fruits and Vegetables

Chop ≥20 lb dense commodities (*e.g.*, potatoes, beets, carrots) or ≥1/2 bushel loosely formed products (*e.g.*, cabbage, lettuce, greens) in a Hobart vertical cutter mixer ≥5 min; stop machine and scrape material by hand into bottom of mixer at least once during operation. This technique was found to produce adequate particle size and distribution in tests with several agricultural products at several time intervals [3].

Hays, Straws, and Dry, Low Fat Feed Ingredients

Grind samples to fine (about 20) mesh in a centrifugal mill using a 1 mm sieve. Collect ground material in the 500-800 g capacity collecting pan and thoroughly mix several batches as necessary to provide appropriate composite from which to take the test portion [4]. Some materials, including hay, may require grinding through a Wiley mill prior to final grinding through the centrifugal mill.

If a centrifugal mill is not available, grind samples through a Wiley mill or equivalent, taking care to prevent physical separation of the product in the mill. A stepwise grinding procedure, with finer grind produced at each step, may be necessary with some products. At each step, grind sample, divide ground material into four sections, reset mill to produce finer particles, and regrind material from two opposite quarters, until final portion is ≤20 mesh.

If the product is to be analyzed for volatile residues, which may be lost because of the heat generated during grinding, cool the mill prior to grinding the sample by grinding dry ice in it.

Oilseeds

Oilseeds are usually hard, as well as high in oil, so special care is required during initial grinding to prevent excessive heat buildup or separation of oil from the rest of the commodity.

Grind well mixed sample in centrifugal mill equipped with 1 mm sieve ring to produce ground product of ≤20 mesh. If noticeable heat builds up, alternatively grind without sieve ring or use sieve with larger openings (*e.g.*, 3 mm), then regrind using 1 or 0.5 mm sieve. Maintain mill rotor speed at 20,000 rpm to aid in cooling.

Dry Products (Pasta, Dry Beans, Grains, *etc.*)

Products such as dry pasta should be treated as described for oilseeds.

References

[1] Wheeler, W.B., *et al.* (1979) *Bull. Environ. Contam. Toxicol.* **23**, 387-390

[2] Thompson, F.D. (Feb. 1976) "Preparation of Fish Sample Composite by Grinding in Frozen State," LIB 1860, FDA, Rockville, MD

[3] More, C.A. (June 1966) "Sample Preparation Using the Hobart Vertical Cutter Mixer," LIB 402, FDA, Rockville, MD

[4] Sawyer, L.D. (Jan. 1977) "A New Mill for Grinding Difficult Samples," LIB 2023, FDA, Rockville, MD

204: SPECIAL REAGENT PREPARATION

204 A: INTRODUCTION

Reagents used in trace analytical chemistry must be carefully chosen and handled to ensure their purity. Impurities in reagents can cause degradation of residues during the analytical process or can cause determinative step responses that interfere with determination of residues. Each laboratory's quality assurance program plan and standard operating procedures (SOPs) (Section 206) should address the way in which reagents are tested for purity, purified if necessary, and stored in a manner that ensures continued purity.

This section contains general tests for reagent and solvent purity. It also provides directions for handling and purifying certain reagents that are common to many methods. Handling and purification of any reagent used in only one method are described as part of that method.

204 B: PAM I CONVENTIONS FOR REAGENTS

Throughout PAM I, the following conventions are used to describe reagents:

1) Lists of reagents for each method specify the grade that should be used; subsequent directions refer to the reagent by name only, unless more than one grade is used in the method.

2) Cross-reference to this section is included in method description reagents lists (Chapters 3 and 4) whenever the method uses a common reagent for which special directions are included here.

3) In almost all cases, solvents must be distilled in all-glass apparatus; in some cases (*e.g.*, HPLC mobile phases), even greater purification is required or recommended.

4) Unless otherwise specified, "water" means distilled water, except where the water does not mix with the determination, as in "water bath." In the latter case, tap water is acceptable.

5) "Ultrapure water," often required for HPLC, refers to the product prepared using the Milli-Q water purification system or its equivalent.

204 C: GENERAL TESTS FOR REAGENT PURITY

Test for Substances Causing Determinative Step Interference

All reagents used in a method should be checked by performing all steps of the method with no sample present. This "reagent blank" should accompany use of any method being used for the first time, or after a period of inactivity, and periodically thereafter. If performance of a reagent blank indicates the presence of an interference, individual reagents (and apparatus) should be examined separately to locate and eliminate it.

Solvents may be checked separately by concentrating 300 mL to 5 mL in a Kuderna-Danish (K-D) concentrator with Snyder column and calibrated receiving flask, each previously rinsed with solvent. Examination of 5 µL concentrated solvent by the determinative step used in the analysis should result in no recorder deflection >1 mm from baseline for 2-60 min after injection.

Other reagents and apparatus used in a method should be checked by rinsing them with solvents that are used in the method, concentrating rinse solvents if appropriate to the method, and checking for responses by the appropriate determinative step. No responses should be detected.

Test for Substances Causing Pesticide Degradation

Substances that degrade residues may also be present in reagents, but these will not be identified by the previous test unless they also cause determinative step response. To detect such impurities, known amounts of chemicals subject to degradation should be added to the extracting solvent (no commodity) and the whole method performed. Noticeable losses of sensitive chemicals indicate the presence of unacceptable contaminants in the reagents. Common contaminants of this type include oxidants that may be present in solvents; these cause degradation of organophosphorus pesticides, especially carbophenothion, particularly during evaporation.

204 D: TESTS AND PURIFICATION PROCESSES FOR SPECIFIC REAGENTS

Acetonitrile

PAM I methods specify use of acetonitrile distilled from all-glass apparatus. To make use of reagent grade acetonitrile, test for impurities by holding moistened litmus paper over mouth of storage container. If litmus paper turns blue, purify 4 L acetonitrile by adding 1 mL 85% phosphoric acid, 30 g phosphorus pentoxide, and boiling chips, then allowing to stand overnight. Distill from all glass apparatus at 81-82° C, discarding first and last 10% of distillate; do not exceed 82° C.

Ethyl Ether

PAM I methods specify use of ethyl ether distilled from all-glass apparatus and assume the presence of 2% ethanol added as a "stabilizer" to prevent formation of peroxides. Practical shelf life is limited, however, even when alcohol has been added; peroxides form readily. Test for peroxides using "Peroxid Test" paper.

Glass Wool

PAM I methods specify use of Pyrex glass wool, which can have contaminants that interfere with determination. If reagent blank tests indicate that glass wool is contaminated, rinse it with solvent and air-dry or heat 1 hr at 400° C.

Some PAM I methods specify silanized glass wool, which may be purchased. To silanize glass wool in laboratory, soak 10 min in 10% dimethyldichlorosilane, rinse with toluene, and soak another 10 min in methanol. Rinse with methanol and allow to air-dry.

Sodium Sulfate

PAM I methods specify use of anhydrous, granular, reagent grade sodium sulfate. To remove phthalate esters that interfere in determinations using electron capture detectors, heat sodium sulfate 4 hr in muffle furnace at 600° C. Store in glass containers; if plastic lids are used, separate them from sodium sulfate with layer of foil.

If reagent blank tests indicate that sodium sulfate is contributing interferences to other determinations, rinse several times with acetone and ethanol, then dry.

Florisil

Florisil, a synthetic magnesium silicate long used as an adsorbent in FDA methodology, is subject to variations in adsorptivity common to most analytical grade adsorbents. Years of experience in using Florisil have led to establishment of procedures for purchasing, handling, and testing the material to optimize and standardize its application. PR Grade Florisil (U.S. Silica, Berkeley Springs, WV 25411) is specified because other grades available from chemical supply companies may be prepared differently by the manufacturer and may exhibit drastically different adsorptivity from what is required for PAM I methods. Handling directions are designed to prevent contamination that may interfere with subsequent analyses and to ensure consistent adsorptivity throughout use of a particular lot of Florisil.

Purchasing and Handling. Observe these procedures for handling Florisil:

- Use PR Grade Florisil, 60-100 mesh, calcined (heated) 3 hr at 1250° F (677° C), for all PAM I methods that require Florisil. Other grades may not be capable of providing the elution patterns required for successful application of the methods.

- Immediately after opening bulk lots of Florisil, transfer to glass containers (preferably amber) that are glass-stoppered or have Teflon-lined or foil-lined screw caps; store in dark. Activate each portion by heating at 130° C for 168 hr (1 wk) before use. Store at 130° C in foil-covered bottles. Florisil may be heated in bulk in pint glass bottles or in individual column amounts in 50 mL Erlenmeyer flasks. Cover containers with foil to prevent contamination, and use in rotation to avoid lengthy storage time. Alternatively, store stoppered container of activated Florisil in desiccator at room temperature and reheat at 130° C after 2 days.

- If entire lot of Florisil is purchased, perform tests below on composite of four-five subsamples taken from each drum with grain trier. Combine subsamples, mix well, and activate mix at 130° C for 168 hr before testing.

Testing. Each lot of activated Florisil must be tested before use to determine whether adjustments in column size are needed to ensure proper elution and quantitative recovery of pesticides. Florisil column size is decreased or increased to adjust for over-retentive or under-retentive Florisil. Two tests should be performed: the "lauric acid test," which measures general adsorptivity, and an elution test that confirms the appropriate elution of pesticides.

LAURIC ACID TEST

Reference

Mills, P.A. (1968) *J. Assoc. Off. Anal. Chem.* **51**, 29-32

Principles

Adsorptivity capacity of Florisil is measured by exposing weighed amount to excess of lauric acid in hexane solution. Amount of lauric acid not adsorbed is measured by titration with alkali. Weight of lauric acid adsorbed ("LA Value") is subsequently used to calculate appropriate weight of that lot of Florisil equivalent to standardized Florisil (LA Value 110).

Apparatus

buret, 25 mL with 0.1 mL graduations, Class A

Erlenmeyer flasks, 125 mL narrow mouth and 25 mL T

GLC, equipped with ^{63}Ni electron capture (EC) and flame photometric, phosphorus mode (FPD-P) detectors (Section 302 DG1, DG2)

pipets, 10 and 20 mL transfer, Class A

volumetric flasks, 500 mL, Class A

Reagents

ethanol, USP or absolute, neutralized to phenolphthalein

hexane, distilled from all-glass apparatus

lauric acid, purified, CP

lauric acid solution, 10.000 g lauric acid/500 mL hexane (1 mL solution = 20 mg lauric acid)

phenolphthalein indicator, 1 g/100 mL ethanol

sodium hydroxide, pellets, reagent grade

sodium hydroxide solution, 0.05 N. Make 1 N solution (20 g/500 mL water), and dilute 25 mL to 500 mL with water. Standardize by weighing 100-200 mg lauric acid into 125 mL Erlenmeyer flask. Add 50 mL neutralized ethanol and 3 drops phenolphthalein indicator; titrate to permanent end point. Calculate mg lauric acid/mL 0.05 N sodium hydroxide (about 10 mg/mL).

Directions

Calculate LA Value for each Florisil lot by performing the following test in triplicate. When method directions in Chapters 3 and 4 require adjustment of Florisil weight for LA Value, calculate as follows: 110/LA Value × weight specified.

- Transfer 2.000 g Florisil to 25 mL T Erlenmeyer flask. Cover loosely with aluminum foil and heat overnight at 130° C.
- Stopper, cool to room temperature, add 20.0 mL lauric acid solution (400 mg), stopper, and shake occasionally 15 min.
- Let adsorbent settle and pipet 10.0 mL supernatant into 125 mL Erlenmeyer flask. Avoid inclusion of any Florisil.
- Add 50 mL neutral alcohol and 3 drops phenolphthalein indicator solution. Titrate with 0.05 N sodium hydroxide to permanent end point.
- Calculate LA Value (mg lauric acid/g Florisil):

$$\text{LA Value} = 200 - \frac{\text{mL required for titration} \times \text{mg lauric acid}}{\text{mL 0.05 N sodium hydroxide}}$$

ELUTION TEST

Reference

Bong, R.L. (1991) Minneapolis District SOPs for Florisil, FDA, private communication

Principles

Solutions of pesticides and butterfat are eluted from Florisil columns, adjusted for LA Value, by eluants from PAM I methods. Appropriate elution of pesticides and weight of butterfat verify that elution pattern and cleanup capacity are adequate. Pesticides are chosen to provide indicators of improper elution, poor Florisil, and impure reagents.

Apparatus

chromatographic column, 22 mm id × 300 mm, Teflon stopcock, coarse porosity fritted disc

K-D concentrator, 500 mL, with Snyder column, two-ball micro-Snyder column, volumetric receiving flask

Reagents

acetonitrile, distilled from all-glass apparatus, see above

ethyl ether, distilled from all-glass apparatus, with 2% ethanol as preservative, peroxide free (see above)

hexane, distilled from all-glass apparatus

methylene chloride, distilled from all-glass apparatus

petroleum ether, distilled from all-glass apparatus

eluants: 6% (v/v) ethyl ether/petroleum ether
15% (v/v) ethyl ether/petroleum ether
50% (v/v) ethyl ether/petroleum ether

eluant 1—20% methylene chloride/hexane (v/v). Dilute 200 mL methylene chloride with hexane. Allow mixture to reach room temperature, and adjust volume to 1 L with hexane.

eluant 2—50% methylene chloride/0.35% acetonitrile/49.65% hexane (v/v/v). Pipet 3.5 mL acetonitrile into 500 mL methylene chloride and dilute with hexane. Allow mixture to reach room temperature and adjust to 1 L with hexane.

eluant 3—50% methylene chloride/1.5% acetonitrile/48.5% hexane (v/v/v). Pipet 15 mL acetonitrile into 500 mL methylene chloride and dilute with hexane. Allow mixture to reach room temperature and adjust to 1 L with hexane.

Pesticide standard solutions, each mL containing these approximate concentrations:

A: 1.0 µg heptachlor, 3.0 µg chlorpyrifos, 2.0 µg heptachlor epoxide, 2.0 µg dieldrin, 3.0 µg endosulfan I, 3.0 µg endosulfan II, 10.0 µg endosulfan sulfate

B: 4.0 µg malathion, 2.0 µg parathion-methyl, 4.0 µg fonofos, 4.0 µg pirimiphos-methyl

C: 20.0 µg Aroclor 1254, 200.0 mg butterfat

D: 1.0 µg α-BHC, 3.0 µg chlorpyrifos, 1.0 µg heptachlor, 2.0 µg heptachlor epoxide, 2.0 µg dieldrin, 2.0 µg endrin, 4.0 µg malathion, 2.0 µg parathion-methyl

Directions

- Prepare three Florisil columns to contain, respectively: 110/LA Value × 20 g, 2 g more than that, and 2 g less than that.
- Rinse columns with 50 mL petroleum ether, discarding rinses. Place K-D with 10 mL volumetric flask under each column.
- Pipet 1.0 mL each standard solutions A and B onto each column. Rinse sides of column with two 3 mL portions petroleum ether, then rinse column with 50 mL petroleum ether.

- Elute each column with 200 mL 6% ethyl ether/petroleum ether. (Collect rinses with this eluate.)

- Change receivers; elute each column with 200 mL 15% ethyl ether/petroleum ether.

- Change receivers; elute each column with 200 mL 50% ethyl ether/petroleum ether.

- Concentrate each eluate, dilute to volume with hexane, and inject about 5 µL into appropriate GLC systems to determine recoveries. Dilute 1.0 mL each standard solutions A and B to 10 mL and use diluted solution as GLC reference standard.

- Consider Florisil lot acceptable if one of three columns permits complete recovery of test compounds and exhibits proper elution pattern (heptachlor, heptachlor epoxide, chlorpyrifos, and fonofos in 6% eluate; dieldrin, endosulfan I, parathion-methyl, and pirimiphos-methyl in 15% eluate; malathion and endosulfan sulfate in 50% eluate; and endosulfan II in both 15 and 50% eluates). Acceptable recovery is >80% for all compounds except heptachlor, and 60-90% for heptachlor. In subsequent use of lot of Florisil, use same weight as that in column with acceptable elution.

- If none of the three columns exhibits proper elution but a consistent relationship exists between weight and elution, test additional columns of weights 3 g above or 3 g below that calculated using LA Value. If these columns also do not exhibit proper elution, it is best to use a different lot of Florisil.

If acceptable weight of Florisil is determined, test that column size further with following procedures:

- Repeat elution tests above, using 1.0 mL each solutions C and D. Elute column with 250 mL petroleum ether, followed by 6, 15, and 50% ethyl ether/petroleum ether eluants; collect each eluate separately. Determine recoveries of pesticides and verify accuracy of elution pattern using gas chromatographic measurement.

- Transfer each eluate quantitatively to separate tared 20 mL beaker. Evaporate solvent on steam bath or hot plate until constant weight is attained to measure amount of butterfat recovered in each eluate. Acceptable lots of Florisil typically permit about 0.3 mg (range 0-1.7 mg) butterfat to elute in petroleum ether eluate, 0.1 (0-0.4) mg in 6% ethyl ether/petroleum ether, 82 (40-135) mg in 15%, and 105 (60-172) mg in 50%.

- Repeat elution tests above, using 1.0 mL each solutions A and B and eluting with Eluants 1, 2, and 3 instead of ethyl ether/petroleum ether eluants.

It is acceptable, once the Florisil lot has been tested and appropriate weight of Florisil determined, to measure and record height of column produced by specified weight; subsequent columns may then be prepared by measuring height rather than weight.

205: REFERENCE STANDARDS

The purity of reference standards and use of appropriate preparation and storage techniques for standard solutions significantly affect analytical results. Reliable and accurate data can be obtained only if correct analytical standard solutions are used for identification and quantitation. Each laboratory's quality assurance program plan (Section 206) should include an element on reference standards and standard solutions. Standard operating procedures (SOPs) should include protocols for obtaining, labeling, storing, and handling standards. This section provides rudimentary information that may be incorporated, as appropriate, into such documentation.

205 A: SOURCES

Reference standards are currently available from several commercial sources, including companies that supply only reference standards, suppliers of specialty laboratory chemicals, and suppliers of chromatographic equipment. Each company publishes lists of reference standards for pesticides, related metabolites, and certain industrial chemicals. Eligible laboratories, mainly Federal Government laboratories, may also obtain reference standards for some chemicals from a repository maintained, under contract, by EPA; eligibility is determined by EPA.

Reference standards in "neat" (undiluted) form, preferably certified by EPA, should be used whenever possible. If neat standards are not available, certified solutions of standards may be used.

205 B: EQUIPMENT AND SOLVENTS

Equipment

Equipment used for preparation and storage of reference standards and solutions includes the following essential, but not all-inclusive, items:

1) analytical balance calibrated for accuracy of ±0.05 mg

2) explosion-resistant refrigerator/freezer, used only to store standards

3) standard solution storage containers:

   a) amber colored, screw-cap bottles, 1 and 2 oz

   b) Teflon-lined caps for bottles

   c) vials for working standards

4) desiccators to store reference standards. Larger vials containing desiccant can be used as individual desiccators for vials of standards.

5) appropriate volumetric glassware, pipets, or microliter syringes

Solvents

Pesticide residue quality solvents are essential for preparation of reference standard solutions. Solvents should be checked before use for the presence of interfering substances by injecting the solvent into the determinative system(s) to be used.

Choice of solvent is sometimes restricted by solubility and stability of the particular chemical. The following solvents, in order of preference, should be used to prepare standard solutions, if suitable for the particular chemical: isooctane (2,2,4-trimethylpentane), hexane, acetone, isopropanol, and toluene.

205 C: STORAGE

Reference standards must be stored properly to prevent undesirable reactions, such as oxidation, re-arrangement, or hydrolysis. Improper storage can lead to loss of integrity of previously acceptable standards. Storage conditions must also prevent the possibility of external contamination. Storage requirements are dependent on the chemical and physical properties of the chemical of interest and are much more stringent for volatile, reactive, or unstable compounds. Review the physical and chemical properties of each compound to determine which storage conditions are appropriate. Minimum requirements for long term storage of analytical reference standards follow:

- If at all possible, store reference standards in tightly sealed containers under desiccation in a freezer.
- Store more stable compounds, such as organochlorine pesticides, in a refrigerator if freezer is not available.

Reference standards that have been stored in refrigerators or freezers must be brought to room temperature in a desiccator prior to weighing.

205 D: PURITY

The analyst is responsible for knowing the purity of the reference standard used to obtain reported data. Follow these rules for recording information about reference standard purity:

- Standards with known purity ≥99%: weight may be recorded as measured; it is not necessary to correct for purity.
- Standards with purity <99%: apply appropriate correction factor to measured weights.
- Technical standards with unknown purity (use only if this is the only available reference standard): record weight as measured, do not correct for purity, but include a note on the source and unknown purity of this standard with the results of any analysis whose results rely on this standard.

205 E: STANDARD SOLUTIONS

Use of inaccurate standard solutions leads to correspondingly incorrect data even if excellent technique and instrumentation are employed. Many analysts consider problems associated with standards and standard solutions as the greatest single source of error in trace residue analysis. Standard solution accuracy is dependent on accurate weighing, correct choice of solvent, chemical stability, appropriate storage conditions, and accuracy in recording the information about solution preparation.

Definitions

The following definitions are used in discussions of standard solutions:

standard "stock" solutions: initial solution from which other dilute solutions are prepared

standard "working" solutions: prepared from stock solutions and appropriately diluted for use in quantitation

Protocols for Preparing Standard Solutions

The following basic requirements are recommended for inclusion in the laboratory's protocol:

Weighing Standards.

- Use only suitable and calibrated analytical balances to weigh standards.
- Weigh solids not affected by moisture on analytical balances with pans open to atmosphere.
- Weigh semisolid standards or liquid standards by technique appropriate to physical and chemical properties of compound. For example, add material to tared volumetric flask, then immediately stopper and reweigh flask.
- Measure appropriate volume of volatile liquid standard in microliter syringe and introduce below surface of solvent in volumetric flask. Dilute to volume. Calculate concentration using volume and specific gravity of liquid standard.

Preparing Solutions.

- Rinse volumetric glassware with solvent in which standard will be dissolved.
- Use pipets, volumetric glassware, or accurate microliter syringes for dilution.
- Dissolve reference standard in solvent in which it is known to be completely soluble. Be aware of any solids remaining in the solution, and ensure that dissolution is complete before using.

- Use solvents of lowest volatility, lowest reactivity, and lowest toxicity possible. If necessary to use a less desirable solvent in order to completely dissolve weighed standard, use minimum amount necessary for complete dissolution, then dilute with solvent of choice. Check for precipitation that may be caused by addition of diluent.

- If possible, verify solution identity and concentration by comparing determinative system data for new solution to data previously reported for that chemical.

Storage of Solutions.

- Be aware that special storage conditions may be necessary for solutions prepared from chemicals that are unstable, reactive, or volatile.

- Prepare new working standard solutions frequently, at least every 6 mo.

- Verify accuracy of concentration of working solutions as needed.

Record-keeping.

- Use similar formats in all types of record-keeping for similar type work. (This approach is also recommended to laboratories within the same organization but at various locations.)

- Record all raw data, such as physical appearance, weight of material dissolved, and dilutions; have all calculations checked by a second analyst.

- Label all solutions with identification number, compound name, concentration, date prepared, analyst initials, and solvent(s) used.

205 F: SAFETY

Procedures for safe handling of reference standards and solutions must be included among the SOPs of each laboratory's Chemical Hygiene Plan (Section 207). Information from the Material Safety Data Sheet supplied with each reference standard and other sources should be used in developing procedures for handling and weighing reference materials. Particular attention should be given to compound toxicity and likelihood of analyst exposure to the compound during handling.

205 G: DISPOSAL OF REFERENCE STANDARDS AND SOLUTIONS

Pesticide reference standards, including primary or working solutions, are classified as hazardous waste under the requirements of the Resource Conservation and Recovery Act. As such, provision for appropriate collection, storage, and disposal of outdated reference standards must be included in each laboratory's hazardous waste disposal plans (Section 208).

206: QUALITY ASSURANCE AND QUALITY CONTROL

206 A: GENERAL PRINCIPLES

Quality assurance (QA) and quality control (QC) should be integral parts of any pesticide residue program in order to ensure the accuracy and appropriate documentation of data generated by the program. The QA process consists of management review and oversight at the planning, implementation, and completion stages of the data collection activity to ensure that data are of the quality required. The QC process includes those activities required during data collection to produce the data quality desired and to document the quality of the collected data. QA activities ensure that the QC system is functioning effectively and that any deficiencies are corrected.

QA/QC programs related to pesticide residue testing emphasize the importance of accuracy and reliability of data. Regulatory action is based on such analyses, as are long term decisions such as banning pesticides. A well-functioning QA/QC program benefits the laboratory and the regulatory program by:

1) ensuring that data are scientifically sound and legally defensible

2) preserving data integrity, validity, and usability

3) ensuring that analytical measurement systems are maintained in an acceptable state of stability and reproducibility

4) establishing the continuing need for training

5) recognizing problems through data assessment

6) establishing corrective action procedures that keep the analytical process reliable

Each laboratory should establish a QA/QC program to ensure reliable analytical data and to document its reliability. This program should include QC procedures, any necessary corrective action, and all documentation required during data collection. The laboratory should prepare, maintain, and support both a written QA Program Plan and standard operating procedures (SOPs). The written QA Program Plan and SOPs should reflect activities as they are currently performed in the laboratory.

Differences between a QA Program Plan and SOPs are subtle. A QA Program Plan provides, in general terms, the QA/QC activities, policies, organization, objectives, and functional guidelines, while SOPs provide detailed step-by-step procedures for operations, analysis, and action. The writing voice for the two types of documents is different, with the QA Program Plans usually written in indicative style and SOPs in imperative style. Written documents (both QA Program Plans and SOPs) that describe procedures performed to accomplish the goals of the program should relate to results rather than to specific activities or procedures. Goals should be achievable and measurable so that the program's success can be evaluated.

Computer applications form a subset of topics within laboratory QA/QC as computers increasingly replace many manual procedures related to laboratory operations and data collection. Computers now manage operations, interface with laboratory equipment, and generate scientific/technical reports. They are increasingly used in laboratories to process, store, and retrieve data; schedule and monitor work throughput; generate test reports; capture data directly from instruments; control critical environmental conditions; and process and display laboratory quality control data. QA Program Plan elements and SOPs should be written to cover computers and their applications wherever appropriate to the laboratory's operation.

This section provides suggestions for a QA Program Plan as well as guidance for preparing SOPs. These materials are suggestions only; their appearance in this manual does not establish them as requirements within FDA or elsewhere. Each laboratory and organization is responsible for preparation of materials appropriate to its own work and required by programs in which it participates.

206 B: QA PROGRAM PLAN

The elements of a written QA Program Plan, outlined below, may be presented in any order. Consideration given to each element will differ among laboratories, depending upon laboratory setting, function, and quality of the measurements deemed essential. A QA Program Plan should fit the needs of the laboratory's program and is not limited to these elements.

Quality Control Points

A. Organizational chart with reporting relationships
B. Responsibilities
   1. Assignments of QC and QA
   2. Management of the quality system

Quality of Equipment

A. Performance criteria for each type of equipment, including computers—minimum standards
B. Responsible person for:
   1. Performance checks
   2. Evaluating performance check results
C. Frequency of performance checks
D. Corrective action—equipment failure
E. Equipment performance log books
F. Equipment maintenance log books—accurate and up-to-date

Quality of Standards and Reagents

A. Preparation, labeling, and documentation of reagents and standards
B. Standards
   1. Identification of primary *vs* secondary standards
   2. Verification of secondary standards (purity, potency, and viability)
   3. Documentation of verification

4. Frequency of verification
5. Handling and storage of standard materials

C. Reagents/media/solvents
1. Handling and storage
2. Procurement procedures to ensure supply and quality
3. Criteria for laboratory water—verification and frequency

Environmental Control/Facility

A. Environmental conditions—documentation
1. Samples
2. Instruments/equipment
3. Computers
4. Personnel

B. Facility
1. Security
a. Laboratory
b. Computer
2. Air handling system—maintenance documentation
3. Sample handling and storage

Quality of Analytical Work

A. Method validation
B. Quality control
1. Responsibility designation
2. Specification of intervals for internal QC techniques
a. Fortified sample
b. Analysis of standard reference material
c. Duplicate analysis requirements
d. Split samples
3. Reference material analysis
4. Corrective action—QC failure
C. Sample analysis procedures
D. Calibration procedure and frequency
E. Corrective action—analysis/calibration failure
1. Decision processes
2. Responsibility for initiation
3. Procedure for correction

Quality of Analytical Documentation

A. Data generation, manual or computerized
1. Data collection procedures
2. Data reduction procedures
3. Data validation procedures

4. Data reporting and approval procedures
   a. Supervisory review of analytical worksheets
   b. Internal laboratory audit of worksheets
   c. Oral review of worksheets
5. Data maintenance (storage, retrieval, and retention)

B. Laboratory notebook and log book policy

Audits

A. Performance audits—internal/external
B. System audits—internal/external

Sample Accountability

A. Sample receipt
   1. Sample custody
   2. Sample tracking
B. Sample storage and handling
C. Sample scheduling
D. Sample disposal/archiving

Quality of Administrative Systems

A. Training program
B. QA reports to management
C. Corrective action procedures

206 C: SOPs

In order to obtain reliable and documented results, adherence to prescribed analytical methodology is imperative. In any operation performed on a repetitive basis, reproducibility is best accomplished through use of SOPs. The SOP describes the commonly accepted method(s) for performing certain routine or repetitive tasks. Adherence to SOPs ensures that analytical results are reliable, reproducible, and properly documented and thus support data quality.

SOPs prepared by laboratories should be up-to-date, comprehensive, clear, and sufficiently detailed to permit duplication of results by qualified analysts. In addition, all SOPs should be:

1) amenable to documentation that is sufficiently complete to record performance of all tasks required by the procedure

2) consistent with current guidelines, regulations, and other requirements

3) consistent with instrument manufacturers' specific instruction manuals

4) inclusive of corrective measures and feedback mechanisms utilized when analytical results do not meet procedural requirements

5) reviewed regularly and updated as necessary when facility or procedural modifications are made

6) capable of demonstrating validity of data reported by the laboratory and explaining the cause of missing or inconsistent results

7) subject to a document control procedure that precludes the use of outdated or inappropriate SOPs

8) available at appropriate work stations

9) archived for future reference or evidentiary situations

SOPs should be written in a format prescribed by the operational QA Program Plan; establishment of a format promotes consistency among SOPs and simplifies the writing process. SOPs are usually written in imperative mode.

Typical topics for SOPs include the following:

1) General laboratory techniques; *e.g.*, use of glassware; glassware cleaning; pipetting techniques; analytical balances, calibration and use

2) Reagents and standard preparation, including source, concentration, storage, and labeling (*e.g.*, see Sections 204, Special Reagent Preparation, and 205, Reference Standards)

3) Sample management; *e.g.*, receipt, handling, and custody; scheduling; shipping requirements; and sample storage

4) Instrument and computer calibration and maintenance; *e.g.*, maintenance logs, procedure and schedule, service arrangements, spare parts

5) Laboratory test methods, including sample preparation and analysis (analyte and matrix specific), test-specific QC, instrument standardization, and quantitation and reporting limits

6) Procedures for data reduction; includes data validation, reviewing, and reporting; verifying electronic data input; and electronic reporting

7) Records management; *e.g.*, generating, controlling, and archiving project-specific and operations records; backup and recovery of computer data; defining raw data

8) Laboratory QC; *e.g.*, procedures for determining method quantitation limits, acceptance/rejection criteria for blanks, matrix-specific quantitation limit, methods precision and bias, matrix-specific bias, matrix-specific precision, control limits for precision and bias, historical performance

9) Laboratory records handling, including review, approval, and revision; computer data entry; and security

10) Waste disposal; includes disposal of samples and waste material

11) QA review; includes requirements for internal, external, and on-site assessment

206 D: BIBLIOGRAPHY

The following references are recommended for guidance in establishing QA Plans and SOPs:

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Garfield, F.M. (1991) *Quality Assurance Principles for Analytical Laboratories*, AOAC International, Arlington, VA

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207: SAFETY

207 A: INTRODUCTION

Chemists analyzing foods for pesticide residues are affected by safety issues, as are all persons working in or managing chemical laboratories. There are many challenges and obstacles to ensuring a safe workplace, especially the costs of upgrading facilities and providing appropriate training, but by developing an effective plan and extensive training of all employees, safe and healthy work conditions are attainable.

It is beyond the scope of the PAM to provide details of an adequate laboratory safety program. Instead, this section is included to provide background information on laws and regulations related to laboratory safety and to stress the need for every laboratory to have a safety plan and for every employee to adhere to that plan. Because the PAM is largely directed toward and used by laboratory chemists, responsibilities of the individual chemist for laboratory safety are emphasized.

207 B: LAWS AND REGULATIONS RELATED TO SAFETY

While it has always been in the best interest of employers to ensure that safety is a primary concern to reduce injuries, property damage, liability, and lost time of employees from the job, occupational safety and employee health issues have drastically changed in the last 20 years as a result of regulatory oversight. The following laws and regulations form the basis for safety requirements in the United States:

1) Williams-Steiger Occupational Safety and Health Act, 1970, which established the Occupational Safety and Health Administration (OSHA) and authorized it to regulate safety and health issues for all employees

2) Executive Order 12196, 1980, which requires each Federal agency to have an occupational safety and health program

3) 29 Code of Federal Regulations (CFR) 1910, which includes OSHA's regulations for toxic and hazardous substances (listed in Subpart Z Sections 1910.1000-1910.1500) that affect laboratory personnel. 29 CFR 1910 includes the following specific regulations:

   a) 1910.1000 establishes, for specific chemicals, limits on the levels to which employees may be exposed. Depending on the hazard of the chemical, a limit is defined either as a permissible exposure limit (PEL), measured in a time-weighted average (TWA) over an 8 hr day, or as a short term exposure limit (STEL), measured in a 15 min TWA exposure, not to be exceeded at any time. Chemicals for which PELs, TWAs, and STELs have been established are listed in Table Z of Section 1910.1000, along with the limits. Implementation of these regulations requires engineering or administrative controls to protect laboratory employees from excess exposure or, in the absence or inefficiency of these controls, use of personal protective equipment (PPE).

b) 1910.1200, also known as the Hazard Communication Standard, requires that manufacturers and importers of hazardous materials protect users from potential dangers by providing written notification of the hazards, in the form of a Material Safety Data Sheet (MSDS) for each chemical. This section also requires manufacturers to provide hazard information on chemical container labels.

c) 1910.1450 establishes the Standard for Occupational Exposure to Hazardous Chemicals in Laboratories ("Lab Standard"). The Lab Standard was issued by OSHA, in recognition of unique characteristics of chemical laboratories, to protect employees associated with laboratory operations. Among other requirements, the Lab Standard requires employers to prepare a written Chemical Hygiene Plan (CHP) to establish work practices, procedures, and policies that reduce the potential for employee exposure to hazardous chemicals. The Lab Standard also establishes requirements for appropriate employee training in safety practices, for monitoring, for appropriate PPE to be worn when hazards cannot be otherwise controlled, and for medical surveillance.

It is FDA policy to select operational strategies that foster a safe and healthful environment for all employees and for those communities in which FDA operates. FDA complies with OSHA regulations, including ensuring that its laboratories operate according to the Lab Standard. To that end, each FDA laboratory has prepared a CHP. FDA's Safety and Occupational Health Management Program is described in the FDA Staff Manual Guide, 2130.1 through 2130.7.

207 C: MATERIAL SAFETY DATA SHEETS

An MSDS, required by OSHA's Hazard Communication Standard, provides precautionary information to the user on physical and health hazards of an individual chemical. Its availability enables chemists to make hazard determinations for materials handled in the laboratory and to identify unsafe conditions that may exist. The recognized source of an MSDS for any hazardous material is the actual manufacturer. If asked, manufacturers usually will provide MSDSs for chemicals purchased prior to the Standard. OSHA also recognizes generic MSDSs as a substitute for those of the actual manufacturer when the original MSDS is not available; these may be substituted for older materials manufactured by a company that has gone out of business.

MSDSs are required to provide the following information in brief:

1) label name of the material; manufacturer's name, address, and telephone numbers for emergencies; and further information

2) list of all hazardous ingredients, by chemical and common names, percentage concentration of each hazardous component, and established PELs and/or threshold limit values (TLVs), established by the American Conference of Governmental Industrial Hygienists

3) various physical and chemical characteristics, including boiling point, specific gravity, appearance and odor, melting point, solubility, vapor pressure, evaporation rate, odor threshold, *etc.*

4) fire and explosion hazard data for fire fighters, with such information as flash point, hazardous fire decomposition products, special fire-fighting procedures, flammable limits, and upper and lower explosion limit concentrations

5) reactivity data for stability, incompatibility, hazardous decomposition products, polymerization potential, and conditions to avoid

6) health hazard information with specific information on acute and chronic health effects expected for each route of entry into the body, whether a carcinogen is present, what signs and symptoms can be expected if exposed, what medical conditions could be aggravated if exposed, and emergency/first aid to be administered if exposed

7) precautions for safe handling and use of the material, including procedures for spill cleanup and waste disposal, conditions to avoid when storing the material, and any other precautions for handling the material

8) recommended control measures, such as what PPE should be used when handling the material, whether special ventilation is needed when handling the material, what glove and eye protection is needed, whether any other protective equipment is needed, and what other work or hygienic practices should be followed when handling the material

MSDSs provide the laboratory employee with a ready source of information about chemical hazard, much of which can be used during development of SOPs (below). MSDSs can also be used to accompany shipments of hazardous materials to fulfill the Department of Transportation requirements for information on spill remediation.

Manufacturers often recommend extensive control methods for use of their hazardous materials to avoid any liability, even when the controls address the concentrated form of the material and normal usage involves a diluted product. MSDSs often do not give specific information for waste disposal and other highly regulated areas, because such requirements vary by geographic location. Specific information is usually lacking for exactly which PPE should be used to avoid specification of trade name products or because the manufacturer has not always thoroughly tested various PPEs with the product. The information presented in MSDSs is usually very general, but technical, in nature. OSHA has expressed concern over the quantity and quality of information present in the MSDSs; manufacturers are required to complete each section, even if the only information conveyed is that inadequate testing has been done.

207 D: DEVELOPMENT OF A CHEMICAL HYGIENE PLAN

As required by OSHA's Lab Standard, each laboratory must have a CHP that provides written statements of work practices, procedures, and policies intended to reduce the potential for employee exposure to hazardous chemicals. The CHP must be made available to all employees. The Lab Standard is a performance-oriented standard that can be readily changed to address the current needs of the laboratory personnel; such flexibility facilitates compliance, despite the complexity and diversity of tasks performed in the laboratory. The CHP must identify:

1) standard operating procedures (SOPs), relevant to safety and health, for activities involving use of hazardous chemicals. Each SOP must identify: the exact nature of the hazard, what safety procedures are established to eliminate or reduce the hazard, what personal protective clothing and equipment are needed to protect the employee, what immediate steps will be taken in the event of an emergency or spill, and what steps will be taken to remedy the situation afterwards, such as decontamination of a spill. SOPs should provide procedures for: handling highly specific reactive chemicals; operating equipment whose use poses potential hazards, malfunction, or repair; handling ionizing and nonionizing radiation; handling compressed and high pressure gases; and working with extremely low or high temperatures.

2) criteria used to determine when additional controls are needed to reduce potential personnel exposure, particularly when highly toxic materials are used. Provision must be made for additional protection when a project involves use of highly toxic or hazardous components, reproductive toxins, or carcinogens. Such protection can include isolating the work area, limiting personnel assigned to the project, providing special PPE, requiring decontamination steps, outlining special waste considerations, and providing special emergency safety equipment and containment devices.

3) circumstances under which certain laboratory procedures require prior approval of the supervisor

4) procedures for medical surveillance and consultation when an exposure is suspected, including an exposure assessment

5) procedures for monitoring for any substance regulated by OSHA, if there is reason to believe that exposure levels exceed the action level, or, in the absence of an action level, the PEL level

6) provisions for maintaining individual employee records of exposure monitoring, medical consultations, and evaluations

7) provisions for personnel training and information

8) procedures for evaluating engineering controls, such as fume hoods, to verify proper functioning

9) identification and maintenance of emergency handling equipment, such as fire extinguishers, eye washes, safety showers, fire alarms, *etc.*

10) measures taken to ensure PPE is functioning correctly

11) emergency response and remediation procedures

12) the Chemical Hygiene Officer who will develop and implement the CHP, and other individuals responsible for implementing any portion of it, including phone numbers

13) procedures for storing materials safely. Issues addressed in this section should include classes of chemicals and segregation to ensure compatibility, expected lifetime of chemicals in general, special cases where degradation is common (*e.g.*, picric acid and peroxide formers), and flammability storage issues. Under the Lab Standard, MSDSs must be maintained and labels on incoming hazardous materials must not be removed or defaced.

14) any other issues, such as individual work practices, attire, electrical hazards, and housekeeping, that affect safety in the workplace

There are many references available to assist chemists in preparing SOPs for the CHP. Information in MSDSs is particularly useful for identifying hazards and safe levels of exposure. Many hazardous materials listed in 29 CFR 1910.1000, Table Z, are routinely used in pesticide analytical laboratories. The levels and standards in Subpart Z are an excellent reference for laboratory personnel for preparing SOPs. A comparison of the odor threshold to the TLV or PEL values is invaluable information in indicating quickly what practices and controls must be in place when working with certain hazardous materials. Often information presented in an MSDS has legal authority and accordingly is an excellent reference. However, according to 29 CFR 1910.1450 (a)(2)(c), the Lab Standard supersedes the Hazard Communication Standard for laboratory operations; more restrictive limits of exposure may be established in the CHP than are established in the MSDS.

Monitoring is conducted to establish the level to which employees are exposed. The Lab Standard requires that engineering or administrative controls be in place to protect chemists from excessive exposure to hazardous materials. For example, one of the most effective engineering controls is proper ventilation and fume hoods. If necessary, controls may extend to the temporary re-assignment of personnel outside the hazard area. In the case of absence or inefficiency of these controls, PPE must be used to protect the chemists; examples include respirators, special gloves, goggles, nonpermeable lab coats, *etc.*

The bibliography in Section 207 G lists government documents and sources, as well as other publications on safety.

207 E: RESPONSIBILITIES OF THE INDIVIDUAL CHEMIST

Every employee working in a laboratory must comply with the agency/company CHP. Each employee must be familiar with safety requirements for working in the laboratory and must adhere to them; each must handle or process all chemicals safely; and each must wear any safety gear and PPE needed to perform laboratory operations safely. Laboratory employees are ultimately responsible for:

1) identifying unsafe or unhealthy situations that exist in the laboratory and reporting such to a supervisor and to the person responsible for the safety program

2) complying with any safety standards applicable to the employee's job performance

3) developing an awareness of activities that may affect the safety of self, fellow workers, and the general public

4) reporting all accidents, injuries, unsafe incidents, or property damage that occur in the workplace

In order to identify unsafe conditions in the laboratory, employees must make hazard determinations for all materials handled there, based upon available scientific information and information found in MSDSs. The ultimate responsibility for a safe working environment rests with each employee.

207 F: ROLE OF TRAINING

OSHA stresses employee training in all their standards as the key to reducing hazards in the workplace. To be effective, laboratory safety training should be tailored to the specific needs of each laboratory. Situations often vary from one facility to the next, and even among the various laboratory operations within the same facility.

Supervisors are often the most knowledgeable about activities within each laboratory and are responsible for training all employees on proper work practices to safely perform laboratory tasks. Safety and health professionals can train laboratory personnel in selection of the proper PPE for each task, maintenance of such equipment, availability of employee services for prevention and treatment of exposures, procedures to follow in an emergency, and conditions to meet and procedures to follow to improve workplace safety and prevent environmental contamination. Employees must be advised about possible sources of exposure, what adverse health effects may result from exposure, what laboratory practices and engineering controls can reduce hazard and prevent contamination of the environment, availability of medical surveillance and environmental monitoring, and their specific responsibilities. An employee who is trained to recognize hazards, and who understands what work practices, PPE, and controls must be implemented, ensures a safer laboratory environment.

207 G: BIBLIOGRAPHY

Government or Organizational Materials

"Pocket Guide to Chemical Hazards," Department of Health and Human Services, National Institute of Occupational Safety and Health (NIOSH) Publication No. 90-117, U.S. Government Printing Office, Washington, DC

For other NIOSH documents, write to NIOSH, 4676 Columbia Parkway, Cincinnati, OH 45226, or call (513) 553-8287. For information on other occupational safety and health problems, call (800) 35-NIOSH.

For National Safety Council occupational safety and health data sheets on specific hazardous materials, call (708) 285-1121; for a list of these data sheets, call (800) 621-7619.

To purchase safety standards for laboratory operations, contact National Fire Protection Association (NFPA), Quincy, MA 02269, (800) 344-3555, or American National Standards Institute (ANSI), New York, NY, (212) 642-4900.

Other Publications

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208: HAZARDOUS WASTE DISPOSAL

All waste generated in pesticide residue analyses must be disposed of properly to comply with local, state, and Federal statutes. Because each locale is subject to different statutes, each laboratory must develop an individual program. Spent solvents and unused reagents generally constitute most laboratory wastes, but other materials that must be discarded may also be considered hazardous; examples of the latter include broken mercury thermometers, unused samples containing a listed or characteristic waste, and contaminated glassware.

It is beyond the purview of the PAM to provide complete directions for hazardous waste programs; the bibliography in Section 208 G is provided to offer guidance beyond the bare outline presented here. Each laboratory is encouraged to assign individuals as Hazardous Waste Managers and to provide appropriate training to those assigned. The complexities and responsibilities associated with hazardous waste management demand no less.

The following outline recommends an approach to developing a program suitable for proper handling and disposal of hazardous waste. In all cases, it must be supplemented by specific local statutes, be configured to the needs of the individual laboratory, and be managed by trained personnel.

208 A: IDENTIFICATION OF WASTE

The first step in developing a hazardous waste disposal program is to determine what wastes the laboratory discards and which of these are classified as hazardous. Under the Resource Conservation and Recovery Act (RCRA), the Environmental Protection Agency (EPA) establishes regulations for determination of hazards and publishes them in Code of Federal Regulations Title 40, Section 261 (40 CFR 261). Those regulations establish that a material is a hazardous waste if:

1) it is not specifically excluded under 40 CFR 261.4.

2) it is listed in 40 CFR 261.30 Subpart D.

3) it exhibits any of the characteristics of a hazardous waste.

4) it is part of a mixture that includes hazardous waste.

The following provides some additional information about these hazardous waste categories; consult 40 CFR 261 for full details.

Waste Specifically Excluded

Certain categories of waste are specifically excluded from being considered hazardous; examples include domestic sewage and household waste.

Chemicals Listed in 40 CFR 261.30 Subpart D

The following lists are published by EPA in 40 CFR 261.30 Subpart D to define those chemicals it classifies as hazardous waste:

F List: Hazardous wastes from nonspecific sources, *i.e.*, generically named wastes. Many F List solvents are used in pesticide analytical laboratories, *e.g.*, "spent halogenated solvents" like methylene chloride and "spent nonhalogenated solvents" like acetone and ethyl ether.

K List: Hazardous waste from specific sources, *e.g.*, "still bottoms from the distillation of benzyl chloride." This list does not contain any laboratory chemicals.

P List: Acute hazardous wastes, specifically named chemicals, *e.g.*, carbon disulfide and fluorine

U List: Commercial chemical products, specifically named chemicals, *e.g.*, acetone and chloroform

P and U Lists contain waste solvents and chemicals and include many solvents used in residue analyses. Any residue remaining in a container or in an inner lining removed from a container that previously held the listed waste is also classified as a hazardous waste, with certain exceptions. All regulated residues, plus any soil, water, or other debris from spill cleanups, are also treated as hazardous, as are any formulations in which a chemical from U or P Lists appears as the sole or active ingredient. The latter category includes primary or working analytical solutions. Some pesticides are on the P List; *e.g.*, aldicarb, aldrin, dieldrin, dinoseb, endrin, parathion, and toxaphene are included on the P List and thus classified as acutely hazardous waste.

Chemicals Exhibiting Hazardous Waste Characteristics

The following characteristics are sufficient to categorize a chemical as hazardous waste, according to 40 CFR 261.20 Subpart C:

Ignitability. EPA defines any solid, liquid, or gas as ignitable waste if:

1) it is a liquid, other than an aqueous solution containing <24% alcohol by volume, and has a flash point <60° C (140° F).

2) it is not a liquid and is capable, under standard temperature and pressure, of causing fire through friction, absorption of moisture, or spontaneous chemical changes, or, when ignited, burns so vigorously and persistently that it creates a hazard.

3) it is an ignitable compressed gas.

4) it is an oxidizer as defined in 49 CFR 173.151.

Corrosivity. A solid waste exhibits corrosivity if it is an aqueous waste with a pH <2 or ≥12.5. Nonaqueous wastes are subject to a steel corrosion test to determine corrosivity.

Reactivity. Solid wastes are considered reactive based on extreme instability and the tendency to react violently or explode; they are considered to pose a problem at all stages of the waste management process.

Toxicity. Hazardous waste is classified as having a "toxicity characteristic" if any of 40 specific contaminants can be extracted at levels greater than or equal to those specified in Table I, 40 CFR 261.24 (b), using an extraction procedure known as the Toxicity Characteristic Leaching Procedure (TCLP). Table I contains mostly pesticides, solvents, and heavy metals that EPA considers potentially leachable into soils or groundwater as a result of improper management. Items such as analytical samples, column packing, and extraction solvents are classified as TCLP waste if any material in Table I is extractable at the regulated levels of concentration. Examples of pesticides in Table I include 2,4-D, 2,4,5-T, chlordane, endrin, heptachlor, lindane, methoxychlor, silvex, and toxaphene.

Hazardous Waste Mixtures

In general, mixing hazardous waste with nonhazardous waste causes the entire volume to be regulated as hazardous waste. Similarly, mixing acutely hazardous waste with hazardous waste may cause the mixture to be regulated as acutely hazardous waste.

208 B: CATEGORIZATION OF WASTE GENERATOR

Once wastes are categorized, a determination must be made of what generator classification the facility meets. The following generator classifications, as defined by EPA, are based on quantity and categories of waste generated:

1) Large quantity generators, *i.e.*, facilities that generate >1000 kg hazardous waste or >1 kg acutely hazardous waste per month, or that accumulate ≥1000 kg hazardous waste on-site

2) Small quantity generators, *i.e.*, those facilities that produce >100 kg but <1000 kg hazardous waste or <1 kg acutely hazardous waste per month, or that accumulate <6000 kg hazardous waste or <1 kg acutely hazardous waste at any one time. Most pesticide analytical laboratories are classified by EPA as small quantity generators because they meet the first criterion. If the quantity of waste from the P List (acutely hazardous waste) is >1 kg per month, then the laboratory becomes a large quantity generator.

3) Conditionally exempt small quantity generators, *i.e.*, those that generate <100 kg hazardous waste or <1 kg acutely hazardous waste per month, or that accumulate <1000 kg hazardous waste or <1 kg acutely hazardous waste at any one time. Generator status varies from state to state; *i.e.*, each state may set its own threshold for generator status. It is therefore necessary to contact the state environmental agency to obtain copies of pertinent regulations.

208 C: OBTAINING APPROPRIATE ID NUMBERS

Once the waste generator category is established, the laboratory should contact the environmental agency of the state in which it is located to obtain both state and EPA ID numbers.

208 D: WASTE COLLECTION AND STORAGE PROCEDURES

Laboratory policy should be established to minimize the amount of waste generated. Where practical and safe, solvents should be recycled by distillation and chemicals should be shared with other laboratories. Analytical procedures should be miniaturized when possible to reduce amounts of solvent and chemicals required. To handle the waste that is generated, laboratory procedures for collection, segregation, and storage of different categories of waste must be established and rigorously enforced. Typical operations include:

1) Arrangement for locations and containers in the laboratory. Hazardous and nonhazardous wastes should be kept segregated during storage to avoid increasing the volume of waste considered hazardous. Waste chemicals from the P List should be separated from other hazardous waste to minimize the amount categorized as acutely toxic. Other segregation should be established for recyclable materials and for wastes with different modes of disposal.

2) Disposal of waste in drains. If local statutes permit, some water-soluble waste, *e.g.*, ethanol and methanol, may be poured down a drain. City pollution control and/or local sanitary sewer district must be contacted to determine local regulations.

3) Waste storage prior to disposal. Maximum storage times for waste are dependent on generator classification and distance between laboratory and waste disposal company. Small quantity generators are allowed to store hazardous waste for 180 days on-site without a permit. Generators accumulating waste in containers must comply with Subpart I of 40 CFR 265.170. All storage vessels must be in good condition and labeled "hazardous waste," and the accumulation start date must be marked on each container.

208 E: ARRANGEMENT FOR WASTE DISPOSAL

Numerous firms specialize in waste disposal. The regional EPA office and/or the state environmental office may provide useful information about local waste disposal firms. Some disposal firms provide consulting services that include preparation of manifests and proper labeling of shipping containers, but the waste generator is ultimately responsible for verifying accuracy of all labeling and shipping information. Department of Transportation shipping data and regulations are published in 49 CFR.

208 F: ADDITIONAL MANAGEMENT REQUIREMENTS

Accurate records must be kept of all waste collection and disposal activities, including manifests and biennial waste analysis and exception reports, when required.

All necessary safety procedures (Section 207) must be followed in handling any hazardous waste.

208 G: BIBLIOGRAPHY

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