Samples - Terminology and Processing

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"Samples"

- When is a sample a "sample"? terminology
  sampling uncertainty
- Sample-prep screening
- EAM sample preparation resource

When is a sample a ‘sample’ ?
(in analytical chemistry)

IUPAC. Compendium of Chemical Terminology, 2nd ed. (the "Gold Book"), Compiled by McNaught, A.D. and Wilkinson, A., Blackwell Scientific Publications, Oxford (2006-), Last update: 2014, version 2.3.3, http://goldbook.iupac.org

Nomenclature for sampling in analytical chemistry (Recommendations 1990), Horwitz, W., *Pure Appl. Chem.*, 62, (1990), 1193

Sample

A portion ... selected from a larger quantity of material

... needs to be qualified (e.g., bulk sample, laboratory sample)

The term 'sample' implies the existence of a sampling error

(i.e. results for the portions taken are only estimates of the concentration ... in the parent material)

If no or negligible sampling error, the portion removed is a test portion, aliquot or specimen.

Use qualifier and be judicious when using the word ‘sample’

e.g.,

laboratory sample

sub-sample

test portion, or analytical portion

Can ‘total analytical uncertainty’ be ambiguous?

measurement uncertainty

(and/or)

sampling uncertainty

Risk Analysis

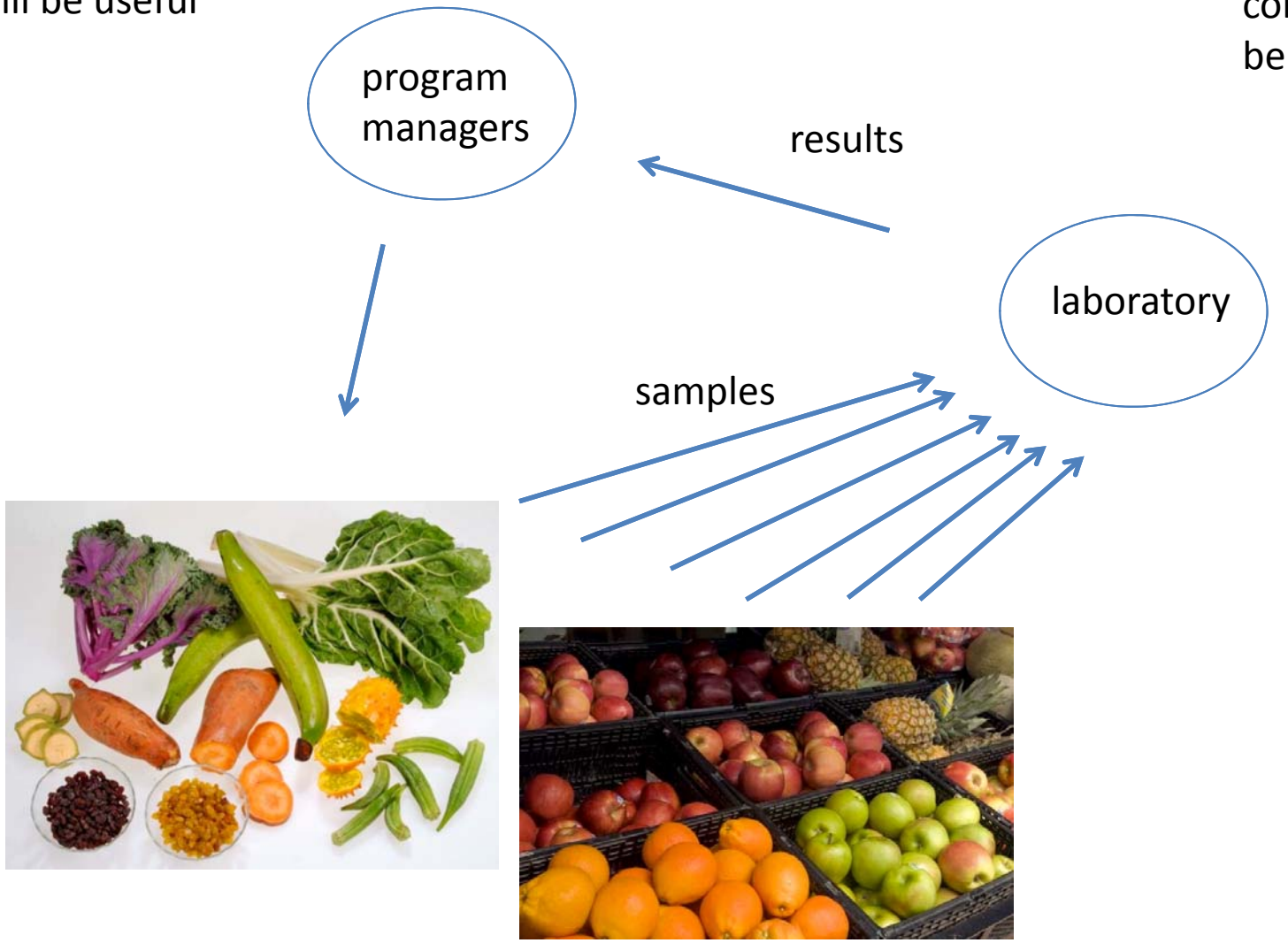
decision making

collect/study the data and information

share info,
address concerns of “stakeholders”

program managers
decide whether screening data will be useful

analysts
determine how composites will be prepared



Sample-prep screening procedure

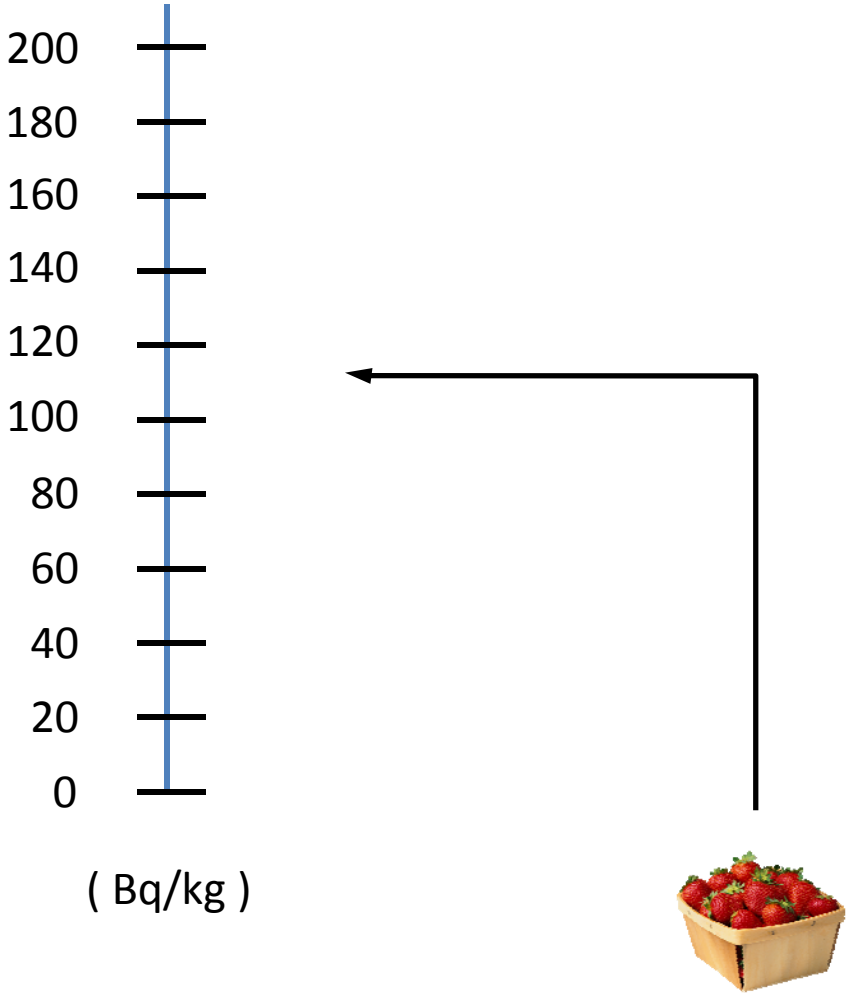
combine/composite *n* sample portions and analyze as usual

but use a “screening level” instead of a DIL

typical analysis

^{90}Sr

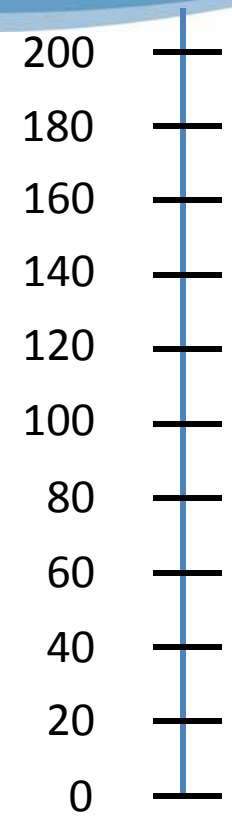
(DIL = 160)



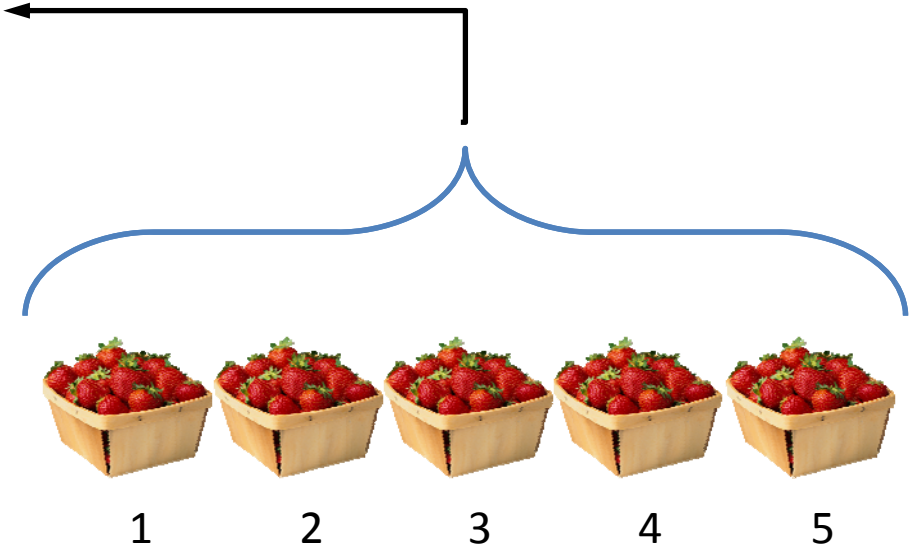
result
below DIL

^{90}Sr screening analysis

(n=5 composite)



(screening limit = 32)



DIL 160 Bq/kg

LOD 0.1 Bq/kg

must be less than DIL / MDL

160 / 0.1 ($n \leq 1{,}600$)

nonhomogeneity

analytical portion 0.1 kg

must use at least 20g / each

100 / 20 ($n \leq 5$)

here, ***n*** is limited to 5 so screening limit = 160/5 = 32

Sample-prep screening procedure

combine/composite *n* sample portions and analyze as usual

use “screening level” (DIL / *n*)

result < screening level - all below DIL

≥ screening level - further analysis

Sample-prep screening procedure

recovery phase; negligible contamination

in conjunction with individual sample analysis

less information but increased sample throughput (β^- and/or α)

Sample preparation resource

FDA's Elemental Analysis Manual (EAM)

www.fda.gov/Food/FoodScienceResearch/LaboratoryMethods/ucm2006954.htm

(www.fda.gov/eam bounces to this address)

2 Sample Preparation

2.1 Food Edible Portion
- 2.1.1 General Procedures
- 2.2.2 Degasification of Carbonated Beverages

2.2 Food Homogenization
- 2.2.1 Laboratory Homogenization Equipment
- 2.2.2 Homogenization Procedures
  - 2.2.2.1 General Procedures
  - 2.2.2.2 Candy Procedures
  - 2.2.2.3 Pills, Capsules, Supplements, etc.

2.3 Digestion and Separation
- 2.3.1 Microwave Digestion (general applications)
- 2.3.2 References to Procedures in Various Methods
  - 2.3.2.1 Leaching Cadmium and Lead from Ceramicware
  - 2.3.2.2 Mercury Separation in Seafood
  - 2.3.2.3 Arsenic Speciation in Rice

2.4 Contamination Control
- 2.4.1 Environmental
- 2.4.2 Laboratory Ware