#### FIFTH PART

### QUALITATIVE APPROACH

Answer to the question "HOW?"

## NUMBER OF DIMENSIONS OF AN OBJECT

#### THREE-DIMENSIONAL OBJECTS:

Strictly speaking all material objets extend in a three-dimensional space.

Practically, this definition is restricted to objects irreducible to a smaller number of dimensions.

Example: 800.000 t lot of bauxite.

- TWO-DIMENSIONAL OBJECTS: when the third dimension, usually vertical, is ...
  - small as compared with the other two and ...
  - pratically uniform.

Model: a sheet of paper.

Examples: a flat truck-load, a forest, a corn field, a flat mineral deposit, a « blister » copper plate etc.

- ONE-DIMENSIONAL OBJECTS when the other two dimensions are :
  - small as compared with the first
  - practically uniform.

Model: cable.

Example: a batch of particulate solids, liquids or pulps **flowing** at a more or less uniform rate. A river. A rail.

Frequent. Easy and cheap to sample correctly.

4

- ZERO-DIMENSIONAL OBJECTS
   By convention: a population made of a large number of units that can have:
  - either a more or less uniform mass
     Example: all manufactured objects.
     Case dealt with by classical statistics.
  - or different masses.
     Example: mineral fragments whose mass can vary in a ratio of 1 to 10<sup>20</sup>.
     Case dealt with by Gy's model (1951).

## CORRECT SAMPLING AND NUMBER OF DIMENSIONS

Experience shows that ...

- The larger the number of dimensions of an object, and the more difficult and costly its correct sampling.
- Correct sampling of irreducible threeor two-dimensional objects is ...

**ECONOMICALLY UNACHIEVABLE** 

## REDUCTION OF THE NUMBER OF DIMENSIONS OF AN OBJECT

• A CORRECT SOLUTION TO THE SAMPLING PROBLEM DOES EXIST ONLY when it is economically feasible to reduce the number of dimensions of a 3- or 2-dimensional object to 1- or 0-dimension, which requires its reclaiming

The economical feasibility obviously depends on the mass to be handled.

- REDUCTION FROM 3 / 2 to 1 dimension The lot is reclaimed and transformed into a flowing stream:
  - particulate solids : at the discharge of a conveyor belt,
  - Iiquids and pulps: at the discharge of chutes or piping systems.

Example: pumping or gravity flow at the bottom of a three-dimensional tank.

- REDUCTION FROM 3 / 2 to 0 dimension the lot is reclaimed and transformed into a population of usually ordered units:
  - particulate solids (trucks, waggons manual/mechanical shovelfuls, etc.)
  - liquids and pulps of ground solids in a liquid (drums, containers, etc.)

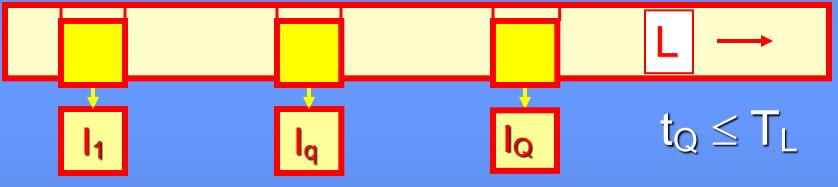
Example (world record, Japan): 16.000 t lot of valuable mineral, reduced to an eight-ton sample processed in the lab.

#### **QUALITATIVE APPROACH**

# HOW TO ACHIEVE A MASS REDUCTION

#### FIRST MASS REDUCTION MODEL

INCREMENTAL SAMPLING
 of a one-dimensional flowing lot:
 Lot L flows from instants t = 0 to t = T<sub>L</sub>

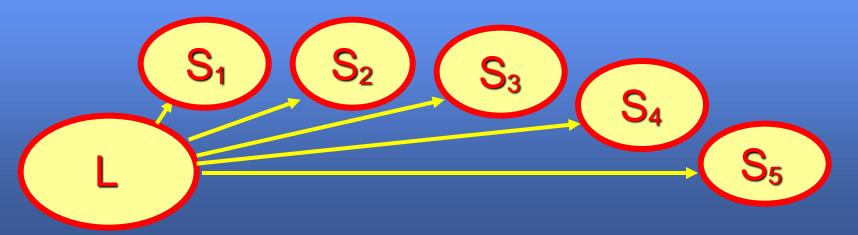


At instants  $t=t_1$ ,  $t=t_2$ ,  $t=t_q$ ,  $t=t_Q$  Q point-increments are selected and extended into Q material-increments  $I_q$ 

Material Sample S is :  $S \equiv \Sigma I_q \ll L$ 

#### SECOND MASS REDUCTION MODEL

• SPLITTING of a zero-dimensional lot. Lot L is split between N twin-fractions that will become N twin potential subsamples. Sample S is obtained by selecting and gathering one or several potential sub-samples  $S_k$ :  $L \equiv \Sigma_k S_k$ 



#### **QUALITATIVE APPROACH**

# THE SAMPLING OF ZERO-DIMENSIONAL MATERIAL BATCHES

## CORRECT SAMPLING OF A ZERO-DIMENSIONAL LOT

Lot L is made of N unspecified units  $U_n$  the mass of which may be uniform or not.

n = 1, 2, ... N • N is large: N >> 1

Q units  $I_q$  are selected : 1 << Q << N. No hypothesis re. a possible correlation between n and properties of  $U_n$  is made.

Sample  $S = \Sigma_q I_q$ . Correctness is achieved by introducing a random factor.

## LOT MADE OF N WELL-DEFINED UNITS OF COMPARABLE MASS

Three selection modes: SY, ST, RA

SY: Systematic sampling with uniform interval N<sub>sy</sub>.

Random selection of increment  $I_1$  in the first interval  $1 \le I_1 < N_{sy}$  .

Then, increment  $I_q$  is defined by :  $I_q = I_1 + (q - 1) N_{sy} \le N$ 

ST: Stratified random.

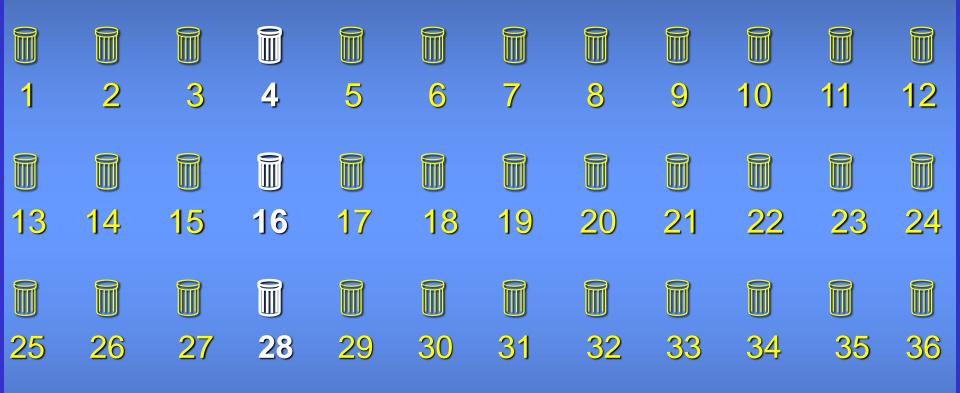
The lot L is first divided into Q strata  $T_q$  of equal length  $T_{st}$ . An increment  $I_q$  is selected at random within each stratum  $T_q$ 

RA: Random.

Q values of q are selected at random between 1 and N, which defines Q increments  $I_q$ 

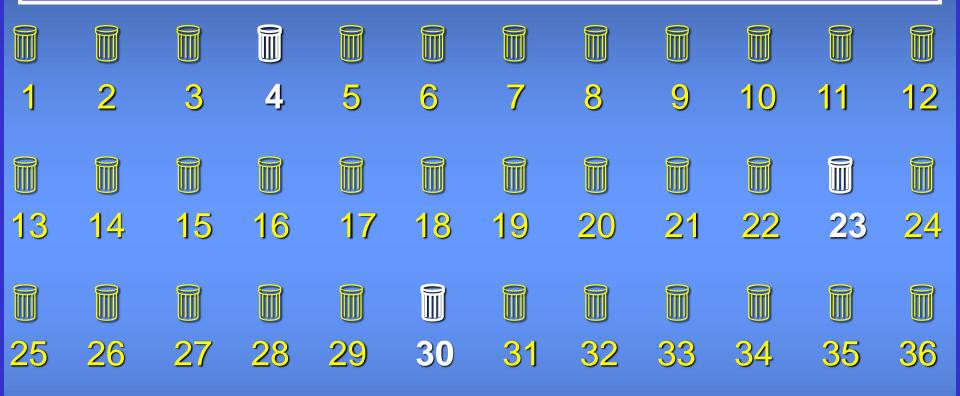
• SAMPLE. In the three cases, sample S is the sum of the Q increments I<sub>q</sub> 16

## SY: L = 756 DRUMS OF RADIOACTIVE RESIDUES COMPARABLE MASS



Lot = 63 groups of 12 drums. Selection of n°4 at random. Interval = 12. S = n° 4, 16, 28

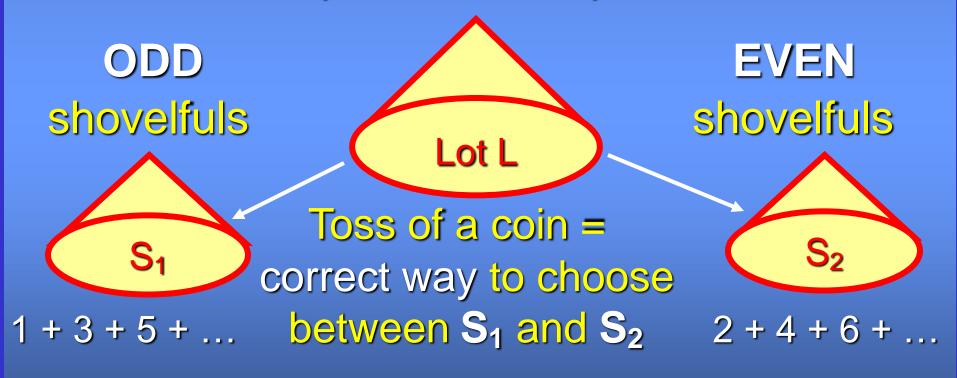
### ST: L ≡ 756 DRUMS OF RADIOACTIVE RESIDUES ♦ COMPARABLE MASS



Lot = 63 strata of 12 drums. S = random selection of n° 4, 23, 30 in strata 1, 2, 3.

## SPLITTING L INTO TWIN-SAMPLES ALTERNATE SHOVELING

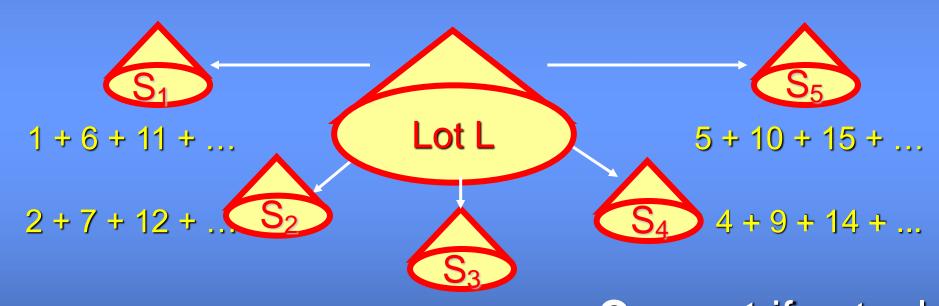
Lot L split by alternate shoveling into  $S_1$  and  $S_2$  = twin potential samples



The operation is usually repetitive

#### **EQUAL FRACTIONAL SHOVELING**

L split by EQUAL fractional shoveling between N twin-samples. Rate 1/N = 1/5

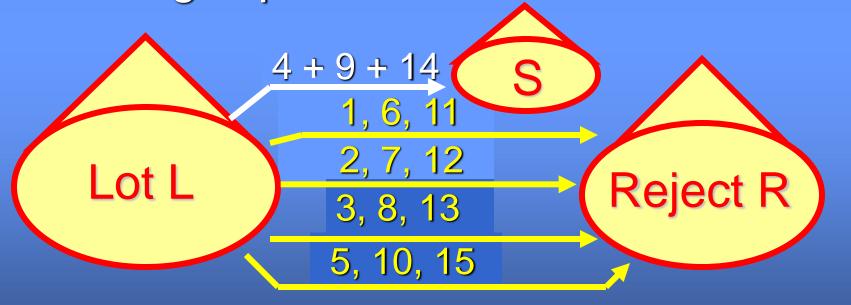


3+8+13+... Correct if actual at random: S<sub>1</sub>/S<sub>5</sub> 20

sample selected at random: S<sub>1</sub> / S<sub>5</sub>

#### **UNEQUAL FRACTIONAL SHOVELING**

L is distributed between two non-twin fractions by UNEQUAL fractional shoveling. This method is NON-PROBABILIST Cheating is possible but can be avoided.



Japan: 16.000 t to 8 t: 1/20; 1/10; 1/10

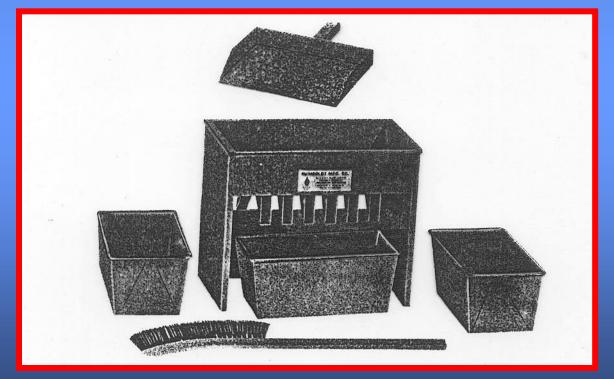
#### THE RIFFLE SPLITTER

Laboratory device meant to split into two « halves » lots weighing (in 2000) up to 1000 kg. Openings from 6 to 80 mm.

 $d_{\text{max}} < 2.5$  to 30 mm

Repetitive operation

Commercial sampling



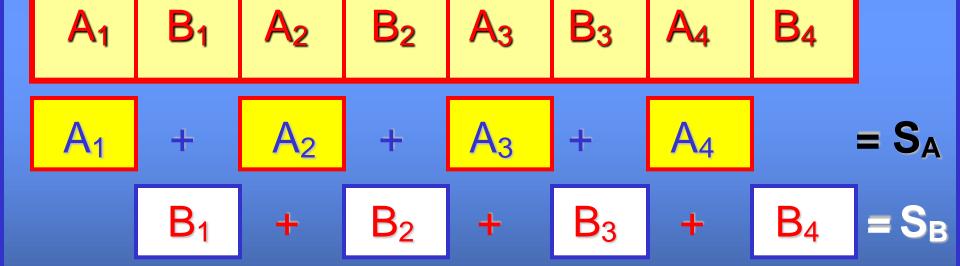
#### THE RIFFLE SPLITTER

Laboratory device. Reliable when implemented correctly (see below). Number N of chutes of a riffle splitter:  $12 \le N \le 24$  with N even Chute width W = 6 to 90 mm. To prevent obstruction: W≥3 x d (fragment diameter). Practically: 2 < d < 30 mm RIFFLE SPLITTERS ARE USUALLY **CORRECTLY DESIGNED** 

Illustrations: Courtesy of Sepor, California

#### PRINCIPLE OF RIFFLE SPLITTING

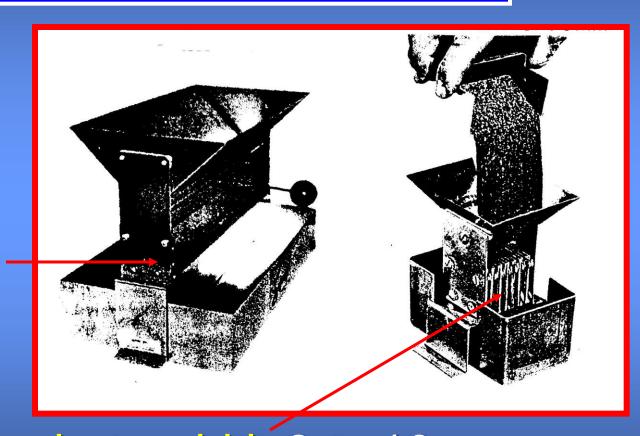
### ZERO-DIMENSIONAL LOT L SPREAD ON A RECTANGULAR SHOVEL



Correct selection of  $S_A$  or  $S_B$  by toss of a coin.

## VARIOUS MODELS OF RIFFLE SPLITTERS

Hopper with gate: allows mixing prior to splitting.



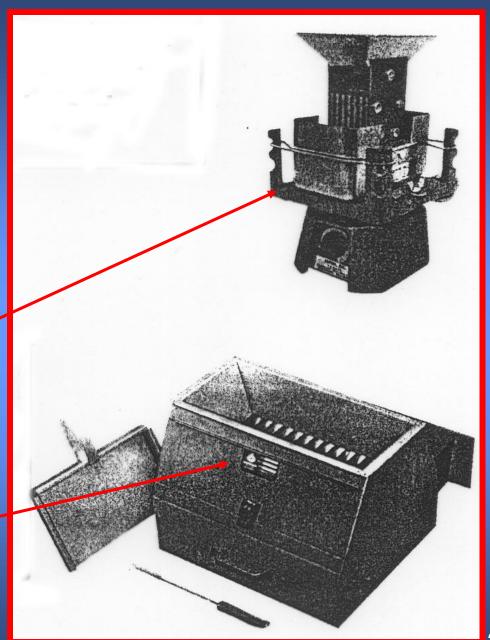
Micro-splitter: chute width 3 to 10 mm

#### RIFFLE SPLITTERS

Dust preventing systems

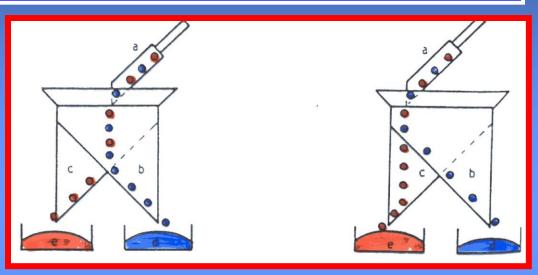
Vibrating

**Enclosed** 



# INCORRECT USE OF A CORRECT RIFFLE SPLITTER

Experiment carried out in 1952



Correct

Incorrect

Four splitting stages → 16 samples

observed biases: G = left; D = Right

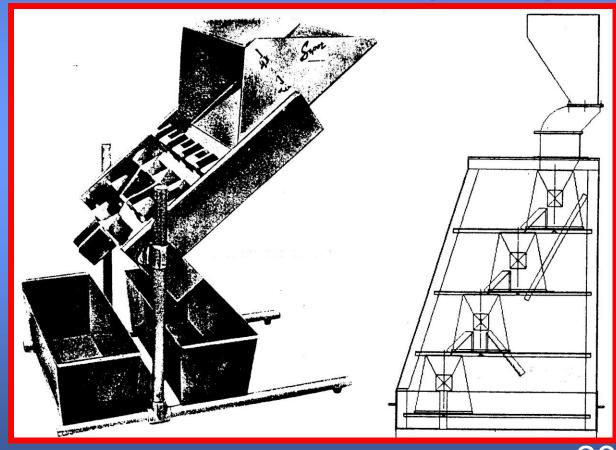
Mass: G > D (6%)  $\Rightarrow$  Pb: D > G (2%)

4 x G 0 x D	3 x G 1 x D	2 x G 2 x D	1 x G 3 x D	0 x G 4 x D
GGGG	GGGD	GGDD	GDDD	DDDD
- 0,201	- 0,051	+ 0,269	+ 0,169	+ 0,214
	GGDG	GDGD	DGDD	
	- 0,221	+ 0,174	+ 0,139	
	GDGG	GDDG	DDGD	
	- 0,341	+ 0,084	+ 0,094	
	DGGG	DGGD	DDDG	
	- 0,281	- 0,251	+ 0,109	
		DGDG		
		- 0,101		
Negative	Negative	DDGG	Positive	Positive
bias	bias	+ 0,189	bias	bias

#### CASCADE OF RIFFLE SPLITTERS

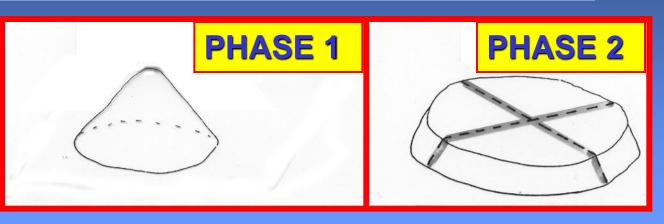
Designed to extract 1/16 in a single pass. This device is INCORRECT in its principle

SHOULD BE
AVOIDED:
SAMPLES
ARE
BIASED

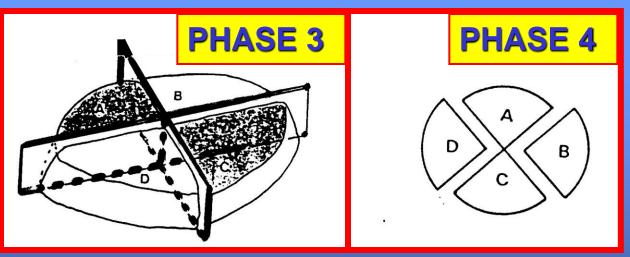


#### **CONING AND QUARTERING**

Uselessly costly hand method



Alternate shoveling always better



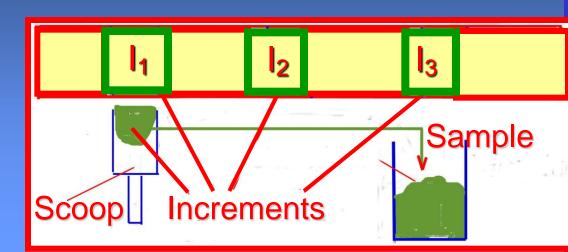
Two potential samples (A+C) or (B+D) 30

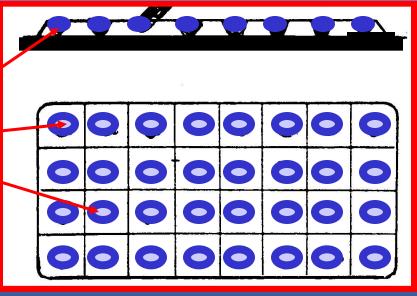
#### THE JAPANESE SLABCAKES

ONE-DIMENSIONAL

Possibly biased

TWO-DIMENSIONAL Increments — Very popular in the Pacific Area

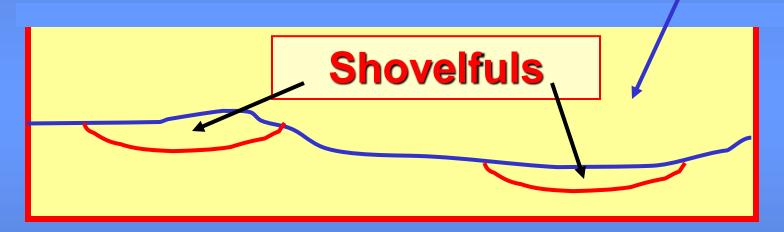




Example: Cattle Food "SAMPLING" = NON-PROBABILIST Picking by means

of a shovel

**Any container** 



One shovelful = one increment Specimen =  $\Sigma$  increments

## SECTORIAL DIVIDER WITH A REVOLVING FEEDER

Usually correct laboratory but also pilot plant device. The feeder F revolves about its axis. Irrespective of its mass, lot L can be divided into N twinfractions. Each of these can be redarded as a 1/N th sample of L.

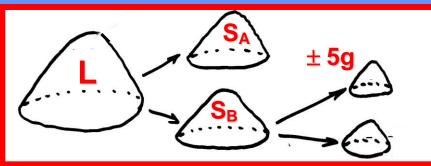
Typically: N = 6, 8, 12. Commercial.

#### **OBTENTION OF A TEST-PORTION**

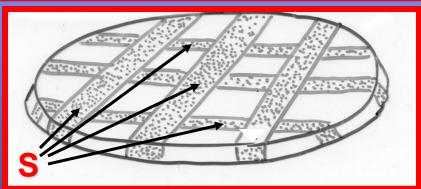
 Use of a rotary divider to obtain a 50-1000 g laboratory sample.
 This is a one-dimensional sampling.



 Alternate shoveling by means of a scoop to obtain 5-50 g



 Mixing and taking the test-portion (e.g. 1 g) by means of a spatula.



#### **QUALITATIVE APPROACH**

# THE SAMPLING OF ONE-DIMENSIONAL MATERIAL BATCHES

## PROPERTIES OF A ONE-DIMENSIONAL OBJECT

l<sub>1</sub> l<sub>2</sub> l<sub>3</sub> l<sub>4</sub> l<sub>5</sub> l<sub>6</sub> l<sub>7</sub> l<sub>8</sub> l<sub>9</sub>

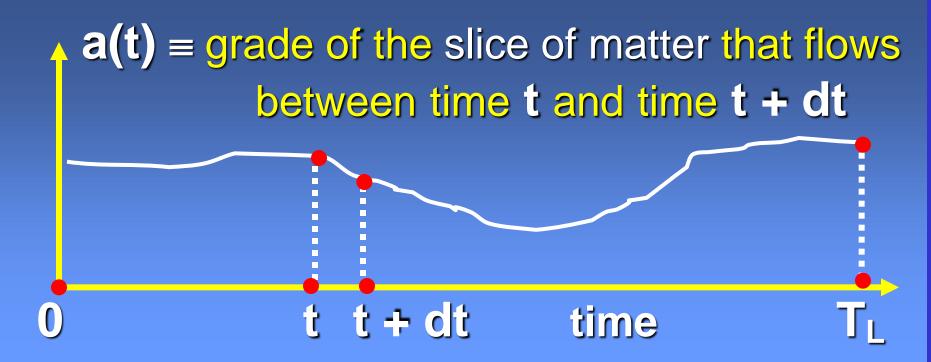
A one-dimensional, compact or flowing object can be regarded as a series of potential increments such as  $I_1 \dots I_9$ . Each increment  $I_q$  may be regarded as a zero-dimensional object, as autocorrelation is barely perceptible on a small scale. Residual autocorrelation generates a 0-dim. Grouping and Segregation Error GSE

#### **AUTOCORRELATION OF A SERIES**

Correlation between two units of a series is called « autocorrelation of the series ». This is the correlation between the properties of increments  $I_q$  (taken at instant  $t_q$ ) and  $I_{q+j}$  (taken at instant  $t_{q+j}$ ) of this series. It is a function of the distance  $j T_0$  ( $T_0$  being the uniform interval).

This autocorrelation gives the series its dimension. Difference with a population.

It is represented by a quasi-continuous curve a(t) ...



Correlation is sensible only in the long range.

On the scale of an increment of length dt or \( \Delta \) it is negligible. For all practical purposes such an increment can be regarded as a zero-dimensional object.

#### THE SAMPLING OF ONE-DIMENSIONAL FLOWING LOTS

- Particulate solids, liquids, gases, fumes Three ways to reduce their mass:
- 1 Taking the totality of the stream during a fraction of the flowing time,
  - Taking a fraction of the stream during the totality of the flowing time,
- 3 Taking a fraction of the stream during a fraction of the flowing time.

1 Taking the TOTALITY of the stream during a FRACTION of the time,

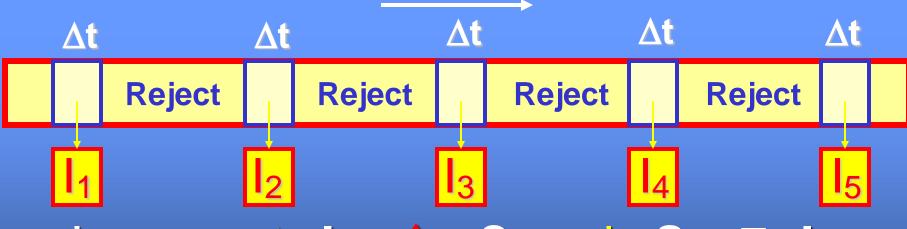
Taking a FRACTION of the stream during the TOTALITY of the time,

Taking a FRACTION of the stream during a FRACTION of the time,



# 1. TOTALITY OF THE STREAM DURING A FRACTION OF THE TIME

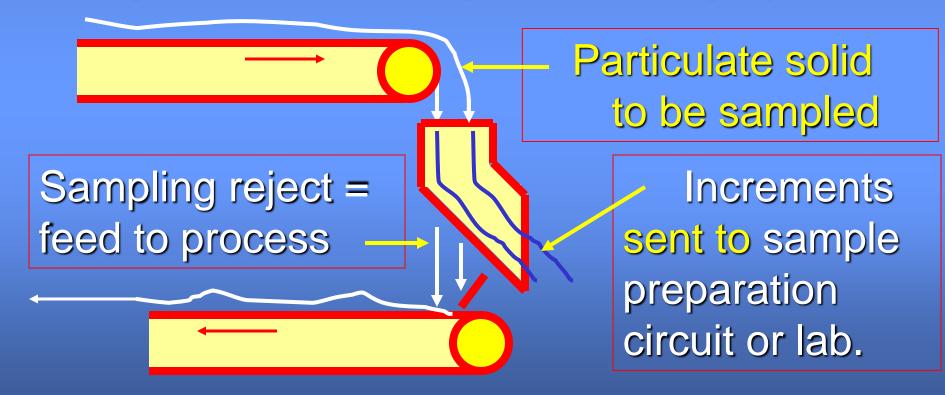
This can be achieved by cutting the stream at a uniform interval  $T_{sy}$  during a certain lapse of time  $\Delta t$ . Principle :



Increments  $I_q$   $\diamondsuit$  Sample  $S = \Sigma_q I_q$  Sampling Reject = Main stream

#### CROSS-STREAM SAMPLERS

Extract the totality of the stream during a FRACTION of the time. Operate at discharge of belt conveyor or piping system.



#### **CROSS-STREAM SAMPLERS**

• STRAIGHT-PATH CUTTER:



Moves between idle positions G and D.

• CIRCULAR-PATH CUTTER:



#### **CROSS-STREAM SAMPLING IS THE**

# ONLY PROBABILIST SAMPLING METHOD

OF ONE-DIMENSIONAL OBJECTS.
FURTHERMORE ...

IT CAN EASILY BE RENDERED CORRECT

# 2 FRACTION OF THE STREAM DURING TOTALITY OF THE TIME

This method is NON-PROBABILIST

S

If there is a correlation between the properties of a constituent and its position in the stream cross-section (e.g. gravity segregation) the specimen S may be heavily biased. Often used in chemical and pharmaceutical industries. 45

#### Implemented in chemical industries

#### LOT L TO BE SAMPLED

Hypothesis of homogeneity throughout the cross-section.

Unreliable Specimen

Practically never observed.

NEARLY ALWAYS BIASED

# 3 FRACTION OF THE STREAM DURING FRACTION OF THE TIME

This method is NON-PROBABILIST



Examples: One shovelful every now and then on top of a belt load. Responsible for a \$7 million cheating over 3 years (tin concentrates). Also used in chemical and pharmaceutical industries.

### FRACTION OF THE STREAM DURING FRACTION OF THE TIME

Example: Cattle Food "SAMPLING":

NON-PROBABILIST Specimen-taking

The paper bag contains nothing but a VALUELESS DANGEROUS SPECIMEN!

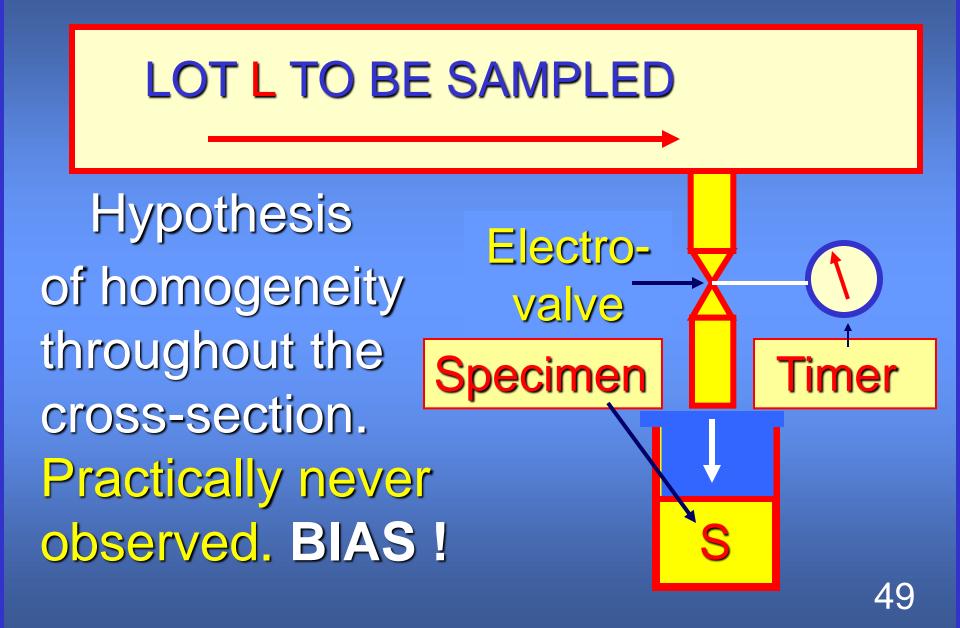
Feed to be sampled

Operator's hand

Paper bag

To conditioning and marketing

#### Example borrowed from chemical industries

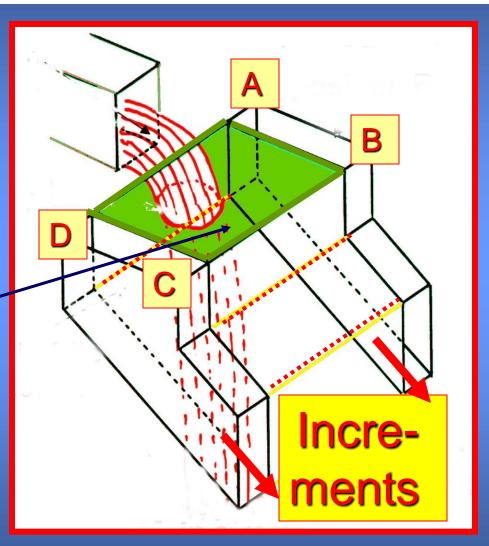


#### THE SAMPLING PLANE ABCD

Straight-path cutter.

Sampling plane

= Area scanned
by the cutter
(green area
ABCD)

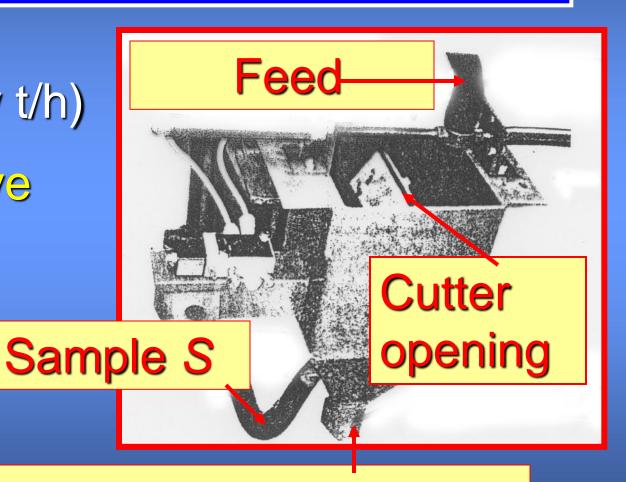


#### STRAIGHT-PATH CROSS-STREAM SAMPLER

Very small flow-rate (few t/h)

Hydraulic drive

Velocity often non-uniform.



Main stream to process

#### REVOLVING SAMPLER (Vezin)

Small to very small flow-rates

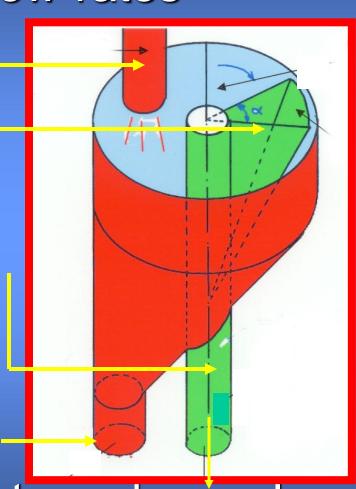
Stream to be sampled

Increment cutter opening

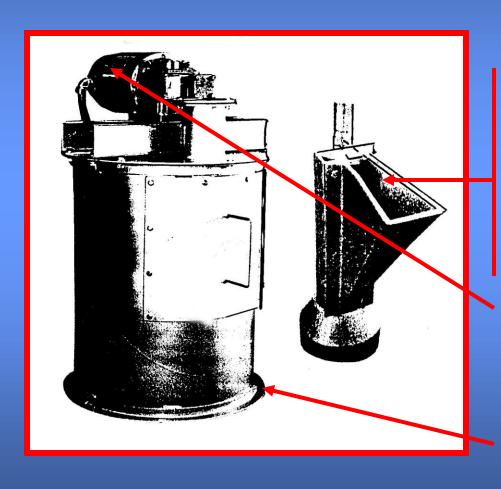
Axial pipe to drive cutter and evacuate increments

Main stream to process

Increments and sample



# REVOLVING SAMPLER (Vezin-type)



Usually correct

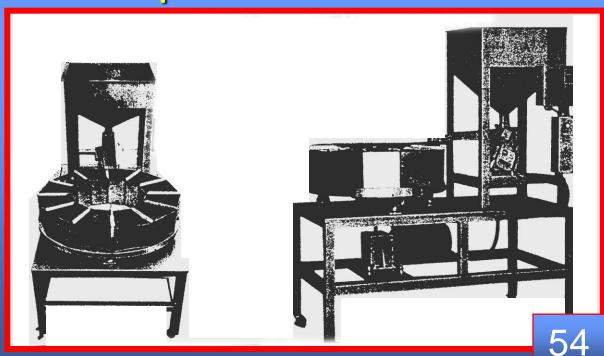
Cutter and axial tube to evacuate increments Electric drive (correct) **External view** 

### REVOLVING SPLITTER WITH A FIXED FEEDER

Turn-table with N removable containers passing through the discharge of a vibrating feeder. Lot L is split between N twin-

samples.

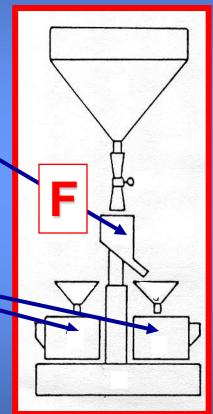
Very small capacity
Commercial operations.



### REVOLVING FEEDER SECTORIAL DIVIDER

Usually correct laboratory but also pilot plant device. The feeder F revolves about its axis, Irrespective of its mass, lot L can be divided into N twinfractions. Each of these can be taken as a 1/N th sample of L.

Typically : N = 6, 8, 12



### DIRECT BOTTLING UP REVOLVING SPLITTER

MINEMET device.

Fixed feeder.

Lot L is distributed between 4 two-liter jars + a reject R.

Completely enclosed dust-proof device designed to split very fine and dry uranate powders.



**Revolving Cutter** 

### DIRECT BOTTLING UP REVOLVING SPLITTER

Same design, smaller scale.

MINEMET device.

Final splitting of uranate powders (Equatorial Africa)
Air moisture and dust perfectly



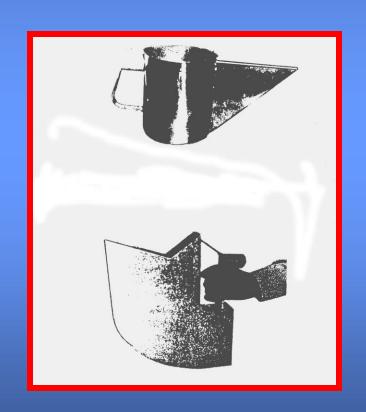
controlled. Feeds  $12 \times 250$  ml jars containing final samples (to parties / reserve). 57

### HAND SAMPLING OF FLOWING STREAMS

#### Incorrect and DANGEROUS instruments

Some of these, have been standardized (!) and are still all too often used at industrial scale.

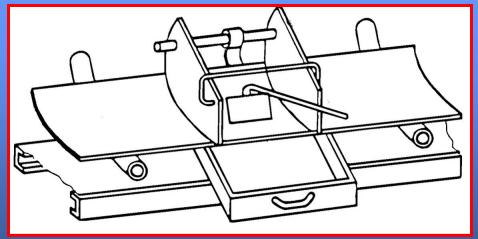
To be avoided whatever the flow-rate.



### STOPPED BELT SAMPLING OF FLOWING STREAMS OF SOLIDS

This hand method is especially implemented as a "REFERENCE METHOD" when testing a sampler for bias. Never implemented industrially.

Correct when the operator does not try to cheat!



## OVERFLOW « SAMPLER » ENSURING UNIFORM FLOW-RATE

One of the most incorrect samplers! The problem was to Feed feed an X-ray fluorescence analyser with a uniform flow-rate of pulp. Hence the overflow tank. **HUGE BIASES** due to differential To X-ray Reject gravity segregation

# HOME-MADE ILL-DESIGNED SPECIMEN-TAKER

#### Nickel mine in South-America

Geological drilling • Biased • Unreliable





#### QUALITATIVE APPROACH

#### THE "SAMPLING" OF TWO-DIMENSIONAL MATERIAL BATCHES

Actually "Non-Probabilist Specimen-taking" 62

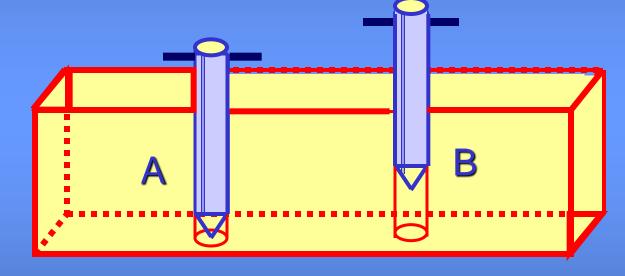
# TWO-DIMENSIONAL SAMPLING **\rightarrow**HAND-PROBE **\rightarrow**

**EXAMPLE**: Truckload of loose sandy





B



Model sample: whole cylinder
Specimen obtained: BIASED

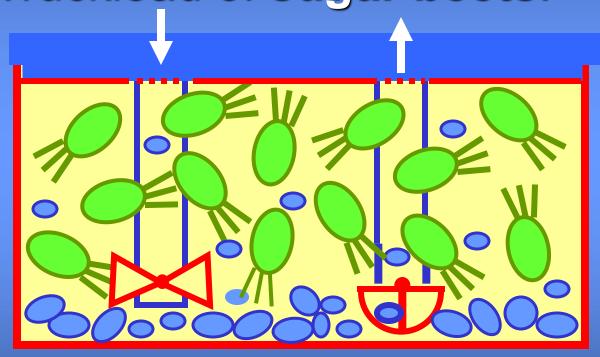
Specimen obtained : BIASED

Practically, never goes to bottom: Ex.

## TWO-DIMENSIONAL SAMPLING MECHANICAL PROBE +



Hydraulically driven probe Flint pebbles deposited on truck bottom by producers



prior to loading. 1000 % bias on SiO<sub>2</sub>%

DELIBERATE DEFRAUDING

#### **QUALITATIVE APPROACH**

# THE "SAMPLING" OF THREE-DIMENSIONAL MATERIAL BATCHES

Actually "Non-Probabilist Specimen-taking" 65

### « SPECIMEN-TAKING » BY VISUAL SELECTION

The operator, a "Sworn Sampler", chooses the fragments he regards as "representative" of the lot.

Actually unreliable "Specimen taking".



#### "SPECIMEN-TAKING" BY MEANS OF THIEVES, PROBES, AUGERS

THIEF: spear-like implement. Textile sacks of coffee, etc.

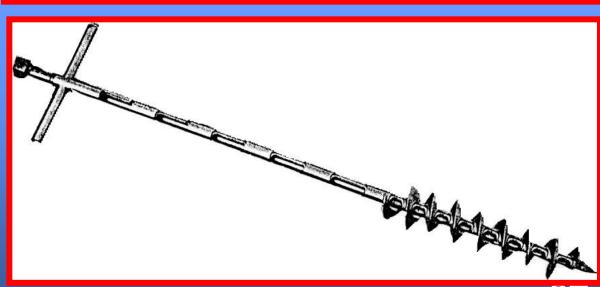


PROBE:

AUGER =

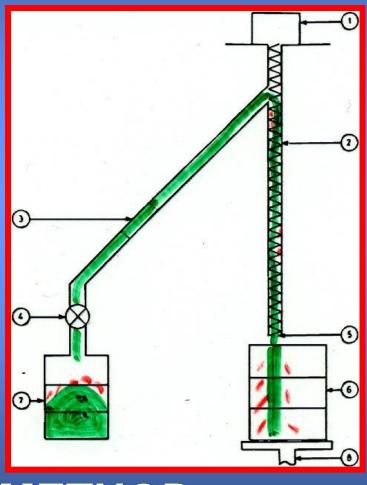
Corkscrew probe





#### "SAMPLING" DRUMS BY MEANS OF A SHEATHED AUGER

Experiment carried out by the U.S. Atomic Energy Commission (1960s) on uranates. Found biased. The drum can be emptied! **CROSS-STREAM** SAMPLING WAS PREFERRED AND

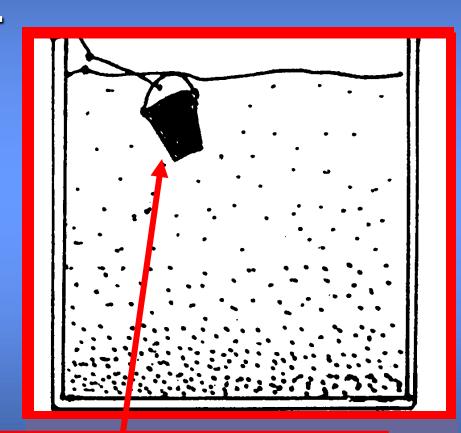


ADOPTED AS A ROUTINE METHOD

### "SAMPLING" A TANKFUL OF A GROUND SOLID « PULP »

#### CHEAP INCORRECT MANUAL METHOD!

Due to selective gravity segregation, the "specimens" are always biased.



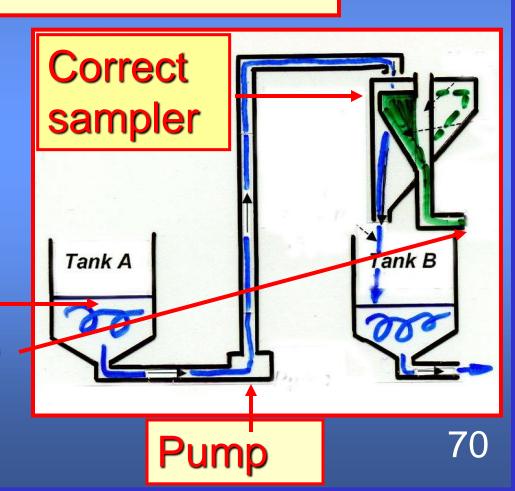
Bucket = specimen

#### « CORRECT SAMPLING » OF A TANKFUL

#### **ONLY CORRECT METHOD**

Lot L is transformed into a one-dimensional object and sampled as such.

Lot **L**Sample **S** 



### « SAMPLING » A GOLD-CYANIDE SOLUTION FOR ITS ANALYSIS

Assayed for Au by Atomic Absorbtion:

The solution was ...

CLEAR: absence of solids / gels

LIMPID: no optical distortion

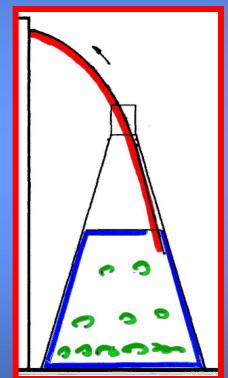
After 11 hours of free settling:

1st reading (no stirring): 387

2nd reading (mild stirring): 550

3 to 8 (violent shaking):  $850 \pm 2$ 

TRAP!



#### **QUALITATIVE APPROACH**

# NON-PROBABILIST SAMPLING SPECIMEN-TAKING

### NON-PROBABILIST SPECIMEN-TAKING

Part of the lot is an unknown quantity

THEREFORE there can be NO THEORY of NON-PROBABILIST SAMPLING

NON-PROBABILIST SAMPLING ERRORS
ARE THEREFORE UNPREDICTABLE

**EXPERIENCE SHOWS they are USUALLY UNACCEPTABLE** 

#### THE THEORY PRESENTED HERE IS A THEORY OF PROBABILIST SAMPLING CORRECT OR INCORRECT

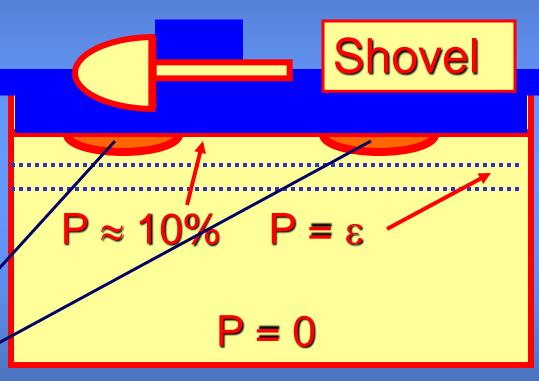
NON-PROBABILIST SAMPLING PROVIDES NOTHING BUT UNRELIABLE SPECIMENS

THESE SPECIMENS ARE DANGEROUS, ESPECIALLY IN COMMERCIAL SAMPLING

#### PICKING = SPECIMEN-TAKING

Picking on top of a container : drum, bag, waggon, truck, etc. Most accessible part of lot L.

Based on hypothesis of homogeneity dangerous because UNREALISTIC.



**Unreliable SPECIMEN** 

# EXAMPLES OF COSTLY SPECIMEN-TAKING ERRORS

- MINERAL PROCESSING PLANT: designed on basis of biased specimens: loss \$10<sup>7</sup> (1960)
- TRADE OF TIN CONCENTRATE: use of method No.3 providing biased specimens ...



Loss of \$7 million over three years (cheating at the smelter).

- BLAST HOLE CUTTINGS: copper-gold mine. Specimen-taking as shown cost the mine \$8 million a year. Two sources of loss:
  - valuable ore was sent to waste pile,
  - waste was sent to plant.

**Specimen** 

A correct sampling plant was amortized in weeks!

