Statens tilsyn for planter, fisk, dyr og næringsmidler

Mattilsynet

Sampling based on the NMKL guide

IAM/MONIQA WORKSHOP, BUDAPEST, SUNDAY 2nd of MARCH 2014

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NMKL PROCEDURE
No. 12 (2014)

GUIDE ON SAMPLING FOR ANALYSIS OF FOODS

CONTENTS

Revised version of NMKL «Guide on sampling for analyses of foods» published in February 2014

- First part: General information on sampling
- Annex – Sampling plans. The Annex is harmonized with CAC/GL 50 - 2004

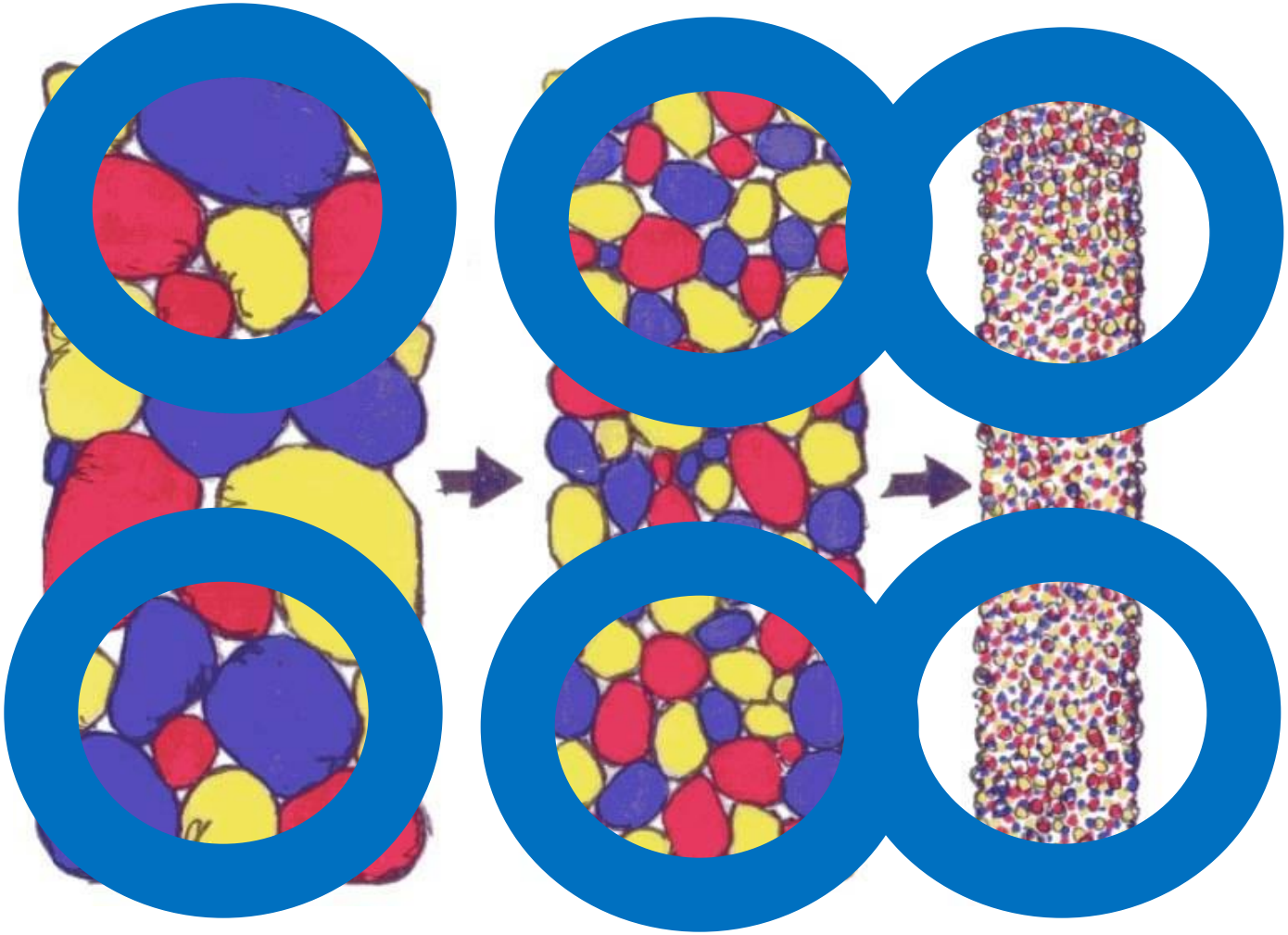
Prior to the sampling ……

- design the sampling procedure carefully
- choose a suitable AQL
- acceptable probability of not rejecting bad lots/rejecting good lots should be considered
- decide where and how to sample
- keep in touch with the laboratory – for specific requirements and delivery
- good sampling protocol – traceability
- decide on transport conditions etc
- use common sense

The characteristics of the parameter and the matrix should be considered:

- any physical, microbiological or chemical changes (sampling to analyses)
- the particle size and the matrix type
- the distribution of the analyte (heterogeneous distribution - more samples needed)
- random sampling/selective sampling

Effects of particle sizes on the sample composition



Heterogenic material, random sampling:

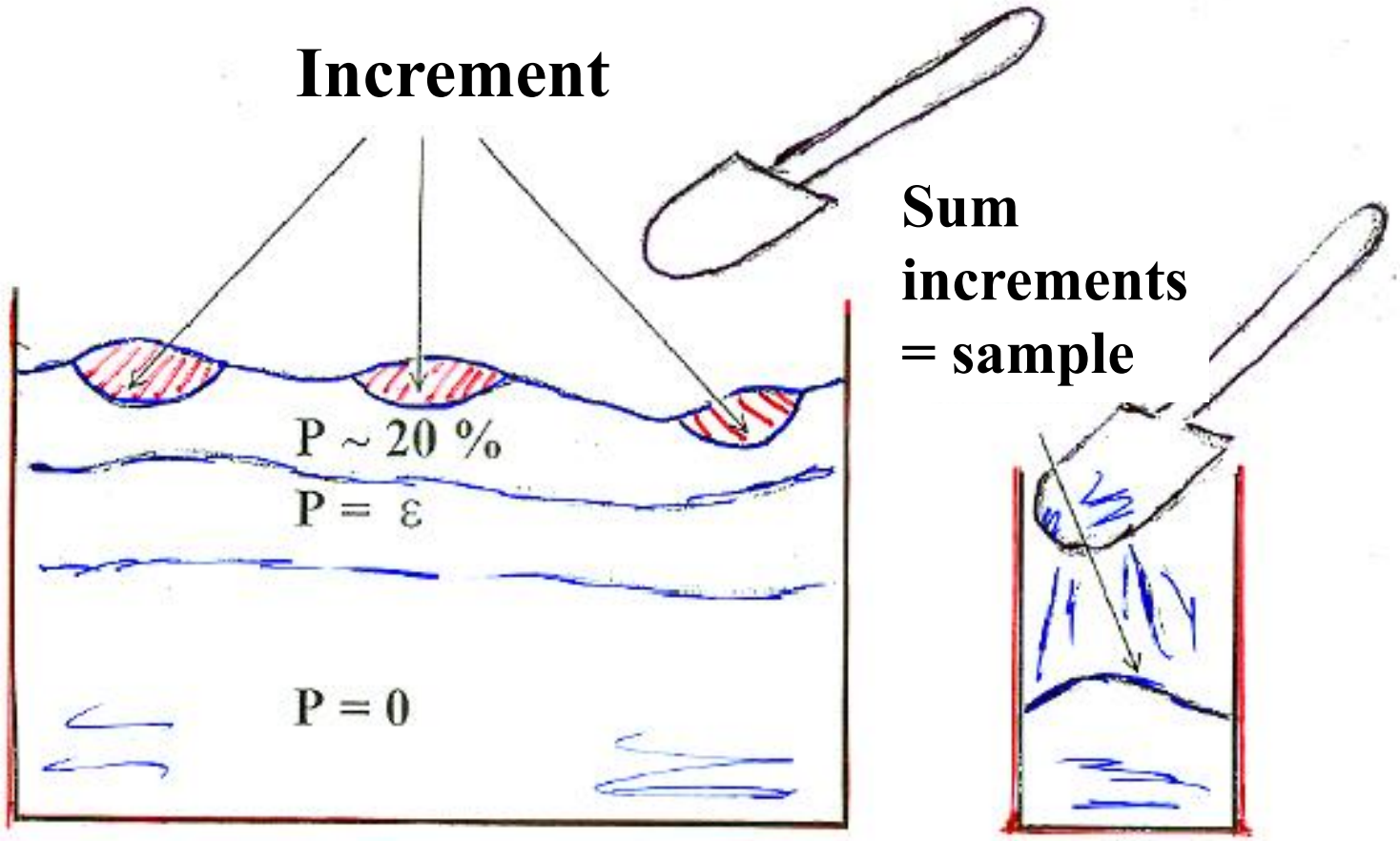
Minimum sample size in gram needed for achieving a reproducibility (with 95% confidence) at ± 2 s = ± 20%

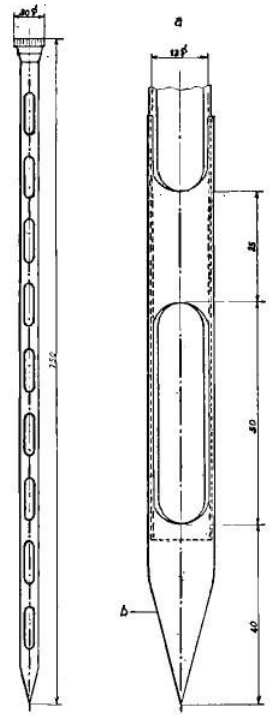
	Level of component in the consignment					
Diameter of Particle	10 %	1 %	0,1 %	ppm	ppb	ppt
1µm	10^{-10}	10^{-9}	10^{-8}	10^{-5}	10^{-2}	10^{+1}
10µm	10^{-7}	10^{-6}	10^{-5}	10^{-2}	10^{+1}	10^{+4}
100µm	10^{-4}	10^{-3}	10^{-2}	10^{+1}	10^{+4}	10^{+7}
1mm	10^{-1}	1	10^{+1}	10^{+4}	10^{+7}	10^{+10}
1cm	10^{+2}	10^{+3}	10^{+4}	10^{+7}	10^{+10}	10^{+13}

From Pierre Gy

Ex.: at ppm level (mg/kg) with diameter of 1 mm: 10 kg sample is needed
at ppb level (µg/kg) with diameter of 1 mm: 10.000 kg(!) is needed.

The probability ”P” for a sample to be withdrawn from the lot





Better to use a probe than a spoon/shovel

Sampling from a stream.

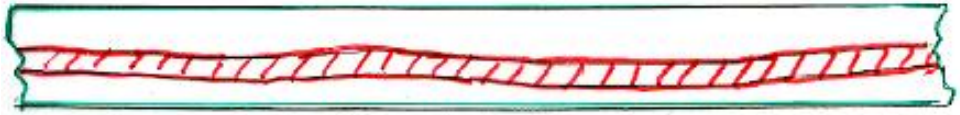
Alternative 1) is recommended

Source: Pierre Gy

0) 2-d projection of stream to be sampled.

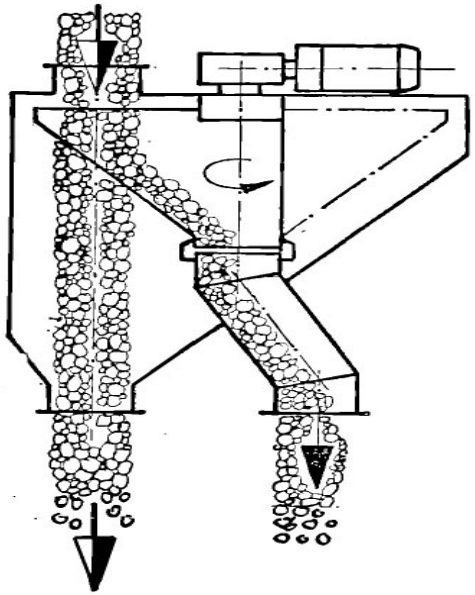
1) Taking the WHOLE OF THE STREAM during A FRACTION OF THE TIME .

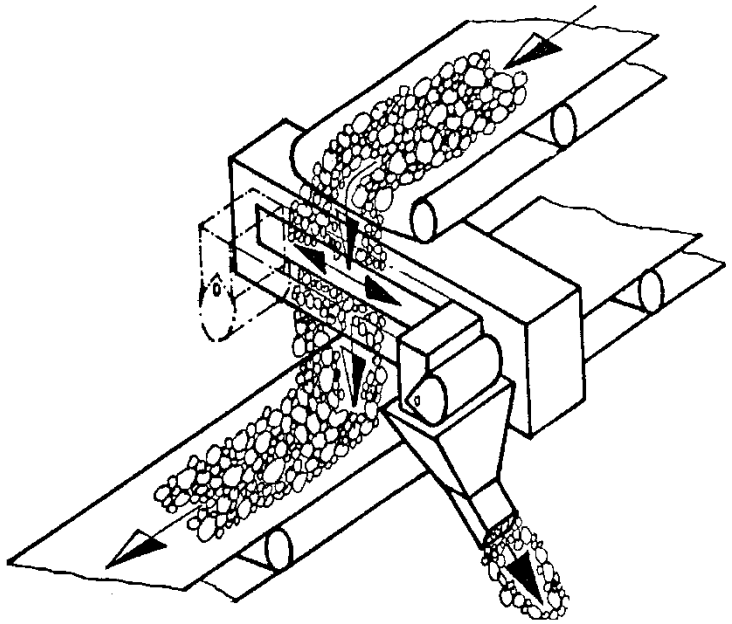
2) Taking A FRACTION OF THE STREAM during the WHOLE OF THE TIME .



3) Taking A FRACTION OF THE STREAM duringA FRACTION OF THE TIME .

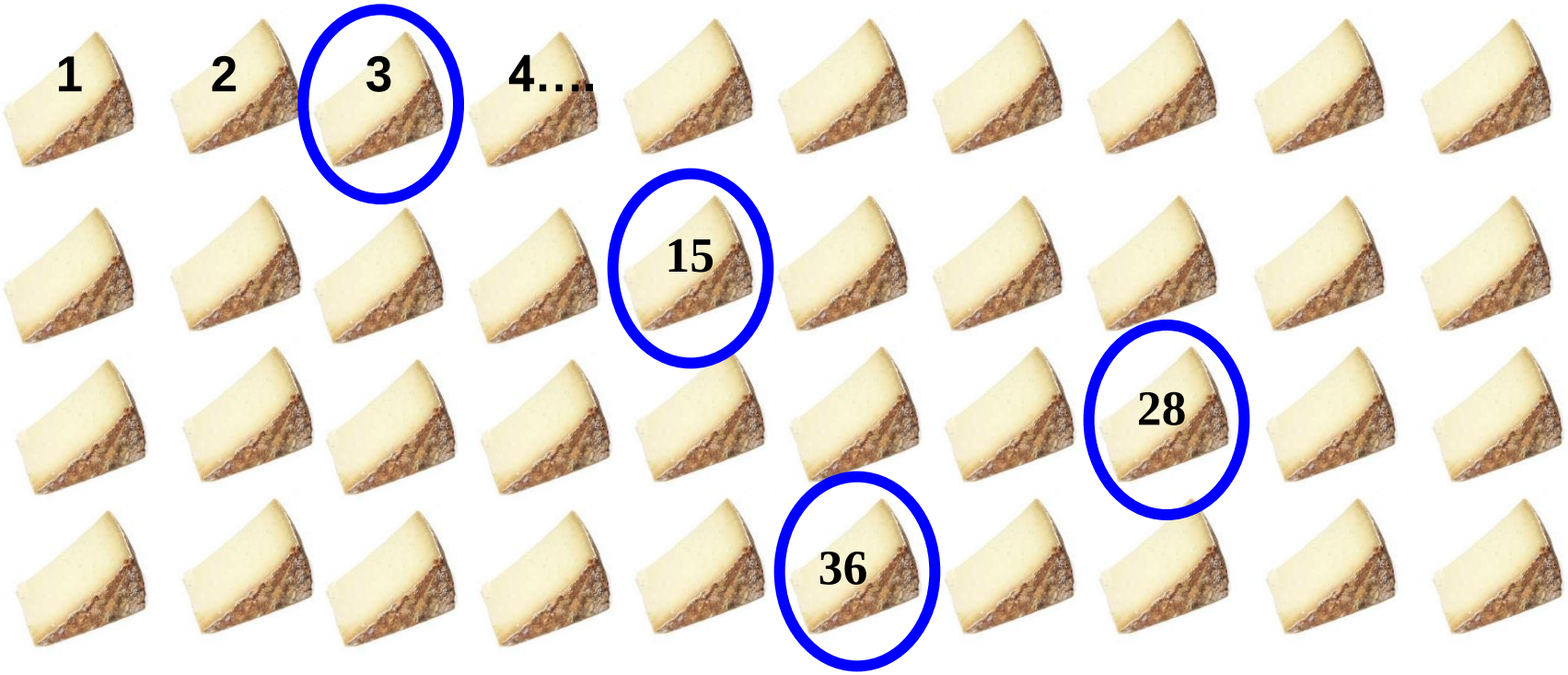
Examples on how to sample the whole stream during a fraction of the time



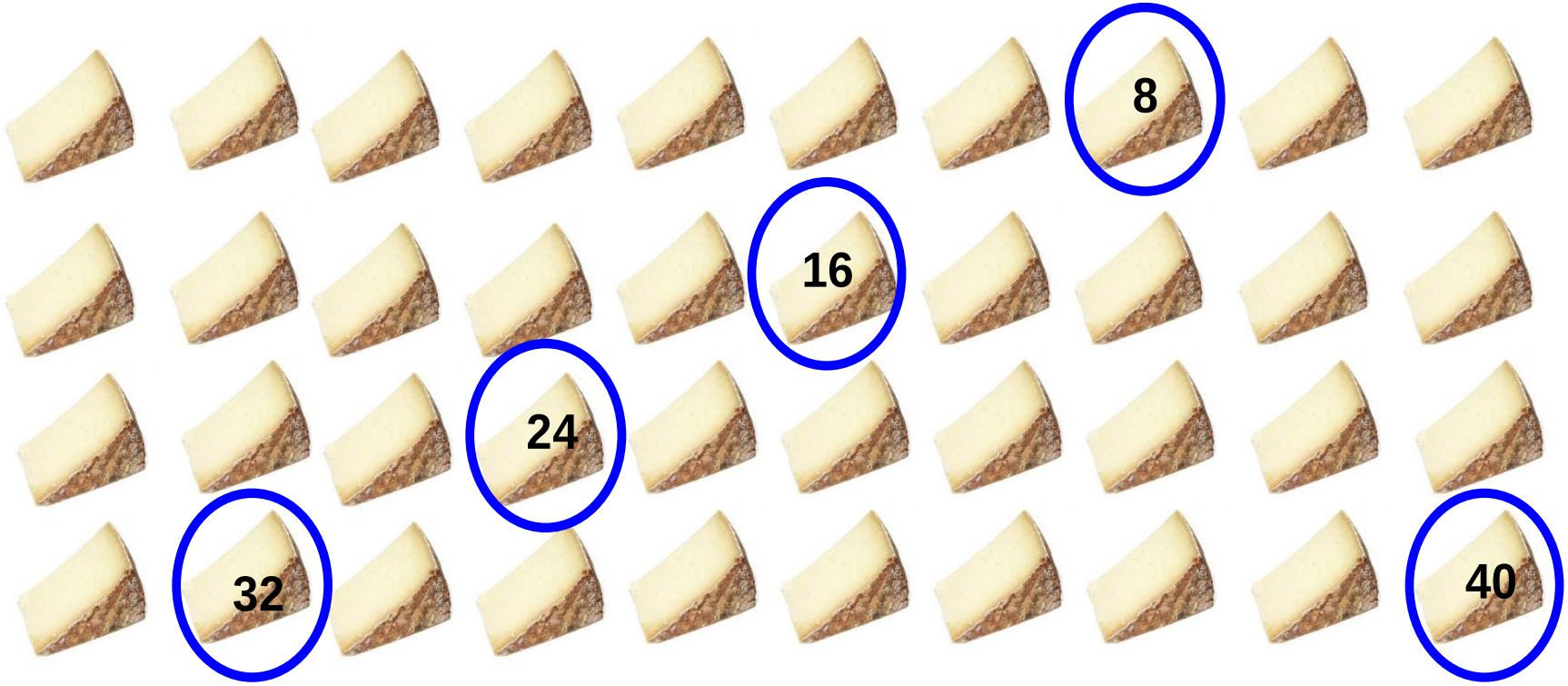


Slit size min 3 x max particle size - the speed
the slit should pas depends on the speed of the stream

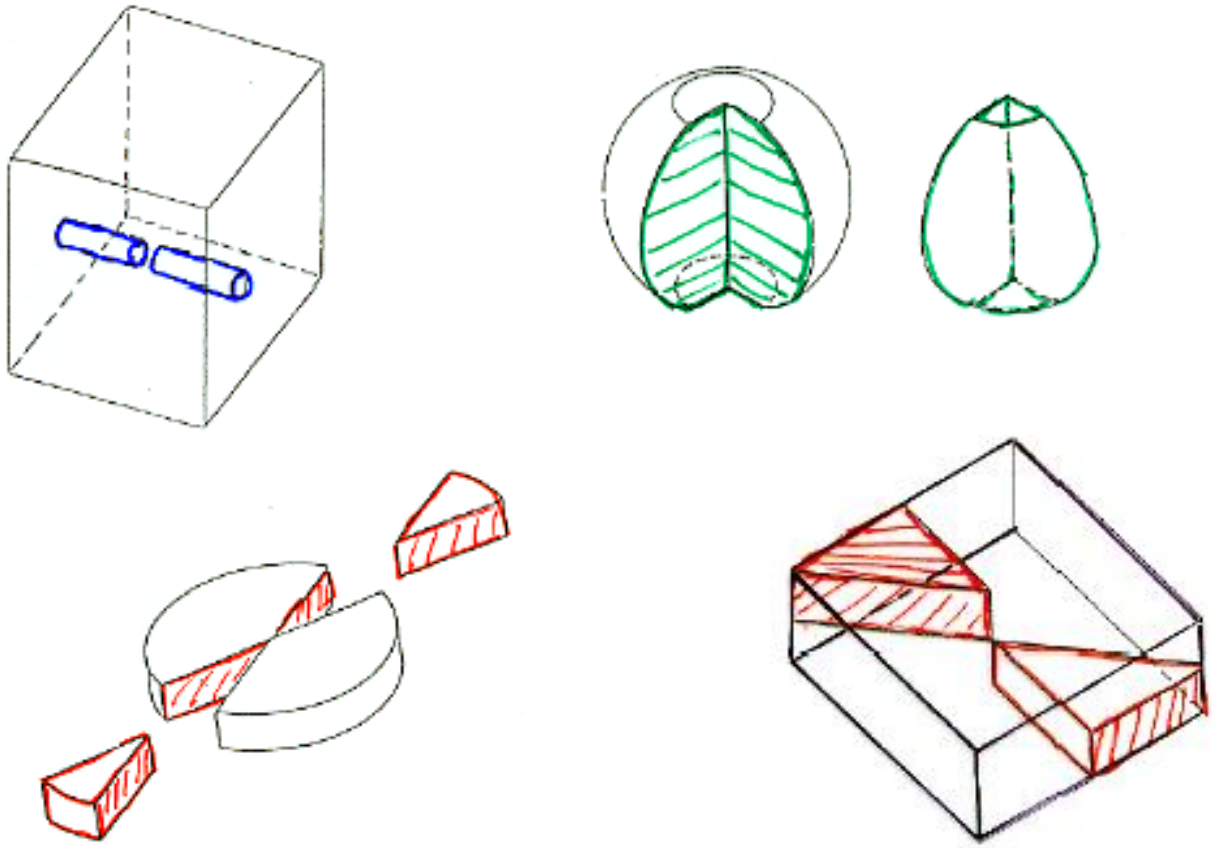
Random sampling



Systematic random sampling

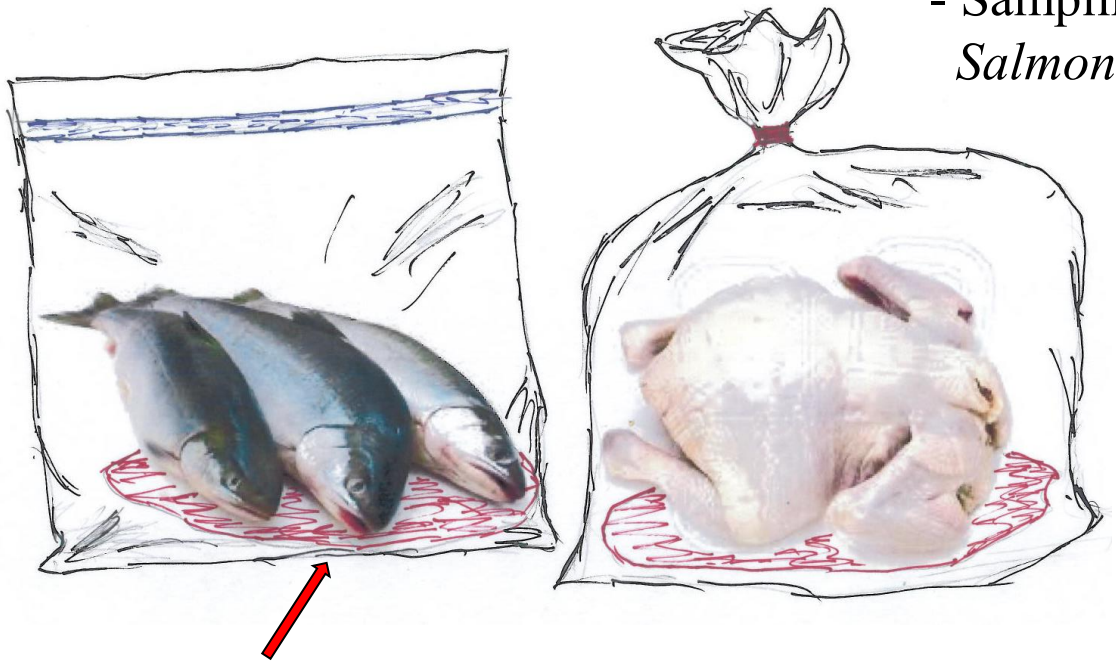


Sampling according to descriptions given in standards: Example from ISO 707|IDF 50



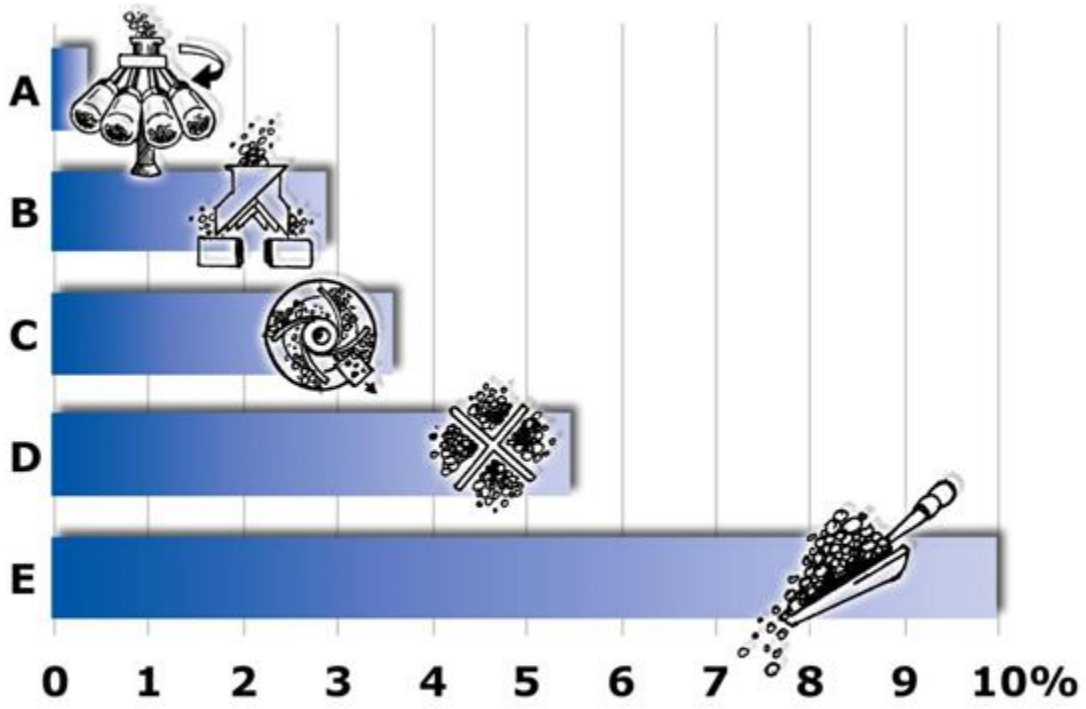
Selective sampling

- Sampling of chicken wings – no *Salmonella* determined.
- Sampling of thawing liquid, *Salmonella* determined



Listeria in fish or fillet of fish: According to observations – higher number of *Listeria* deeper down in a container. Sampling of runoff liquid at the bottom of the container might be the best place of sampling at a quality control in the production of sushi!

Standard Deviations of various sample divisions methods



Sample divider

Sample splitter

Disc divider

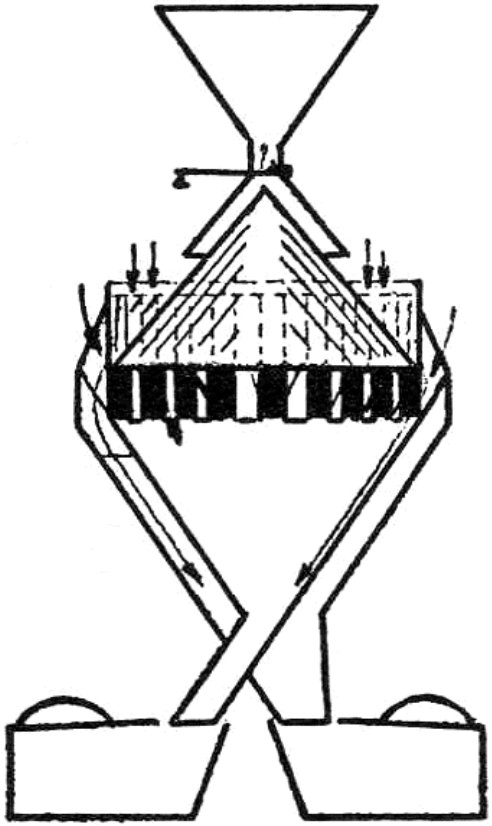
Cone and Quatering

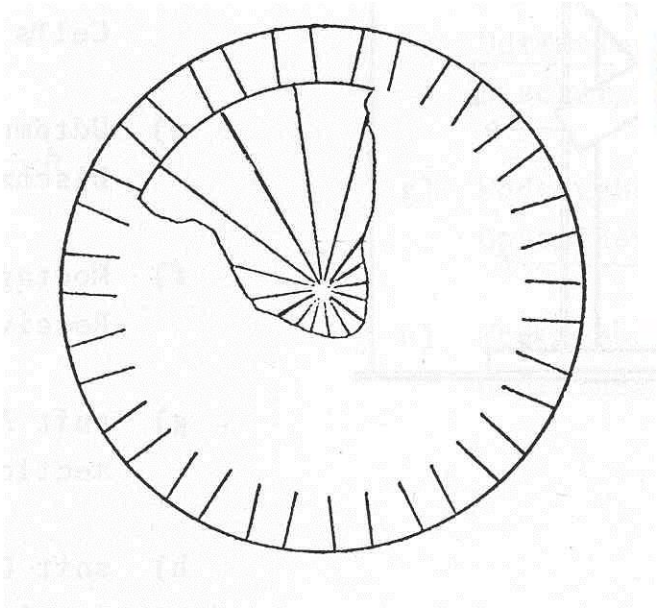
Random sampling, with a shovel/spoon

Material feed, particle size < 5 mm

Source: Retsch

- Sample Splitters: Dividing in two parts -





Source.: NMKL-method no. 34 (withdrawn)

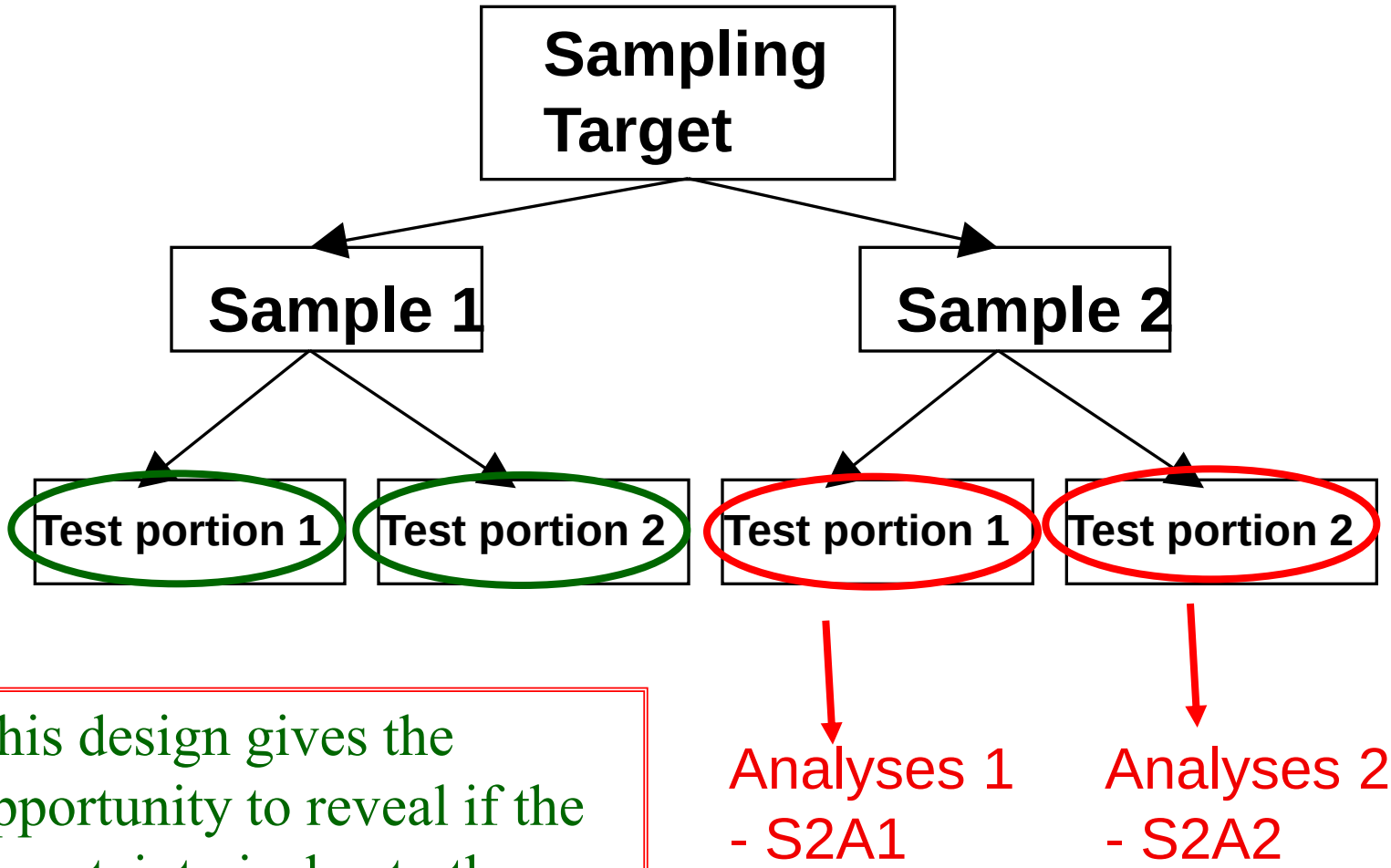
Rotary Tube Divider

Source: Retsch

Slurry milling/homogenisation of large samples (up to 30 kg)

Fig. from www.silverson.com

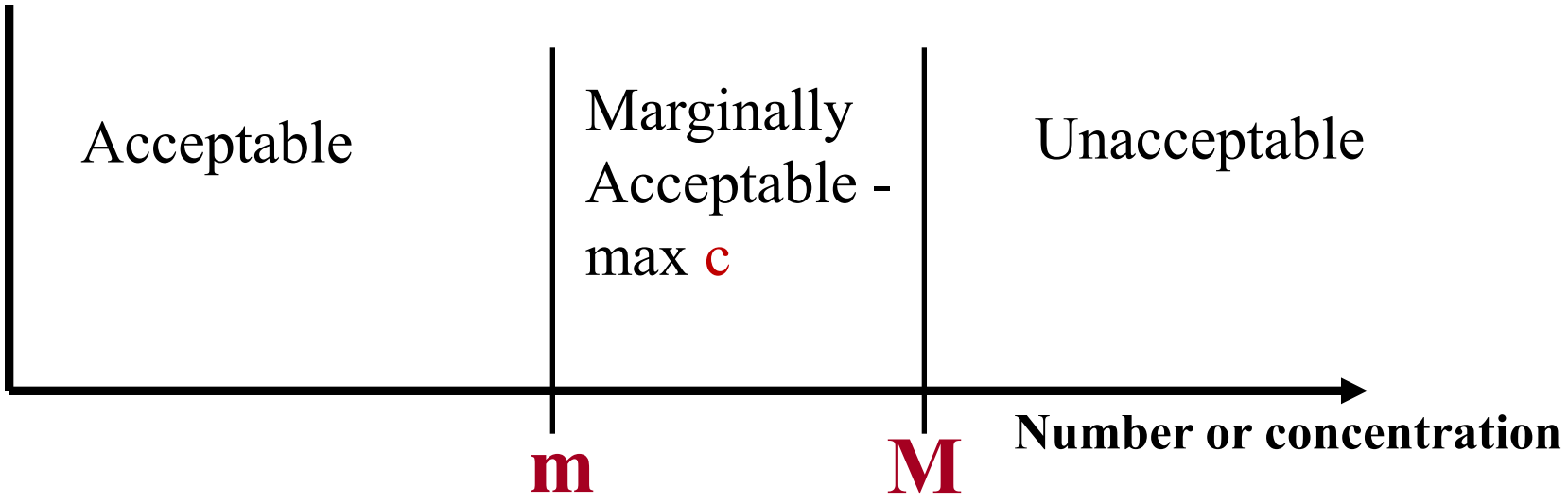
Replicate design with two split levels



This design gives the opportunity to reveal if the uncertainty is due to the sampling or the secondary sampling/analyses

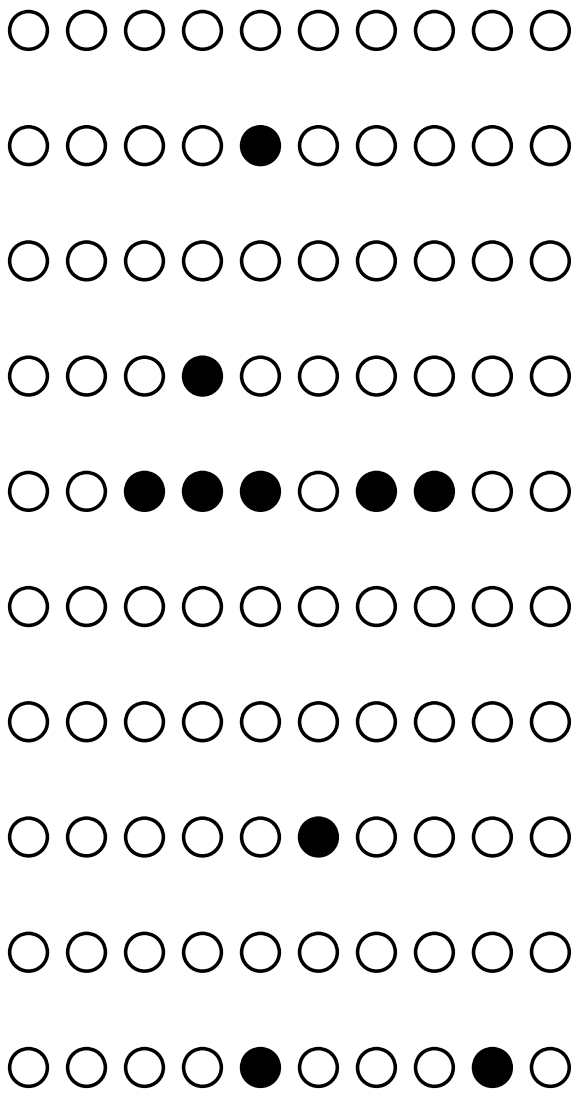
Two- and three-class sampling plans

A three-class sampling plan requires *n*, *c*, *m*, and *M*
- where M the upper limit that must not be exceeded.

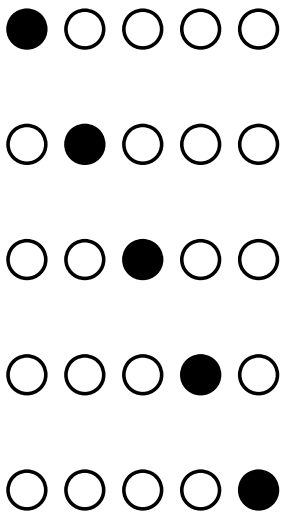


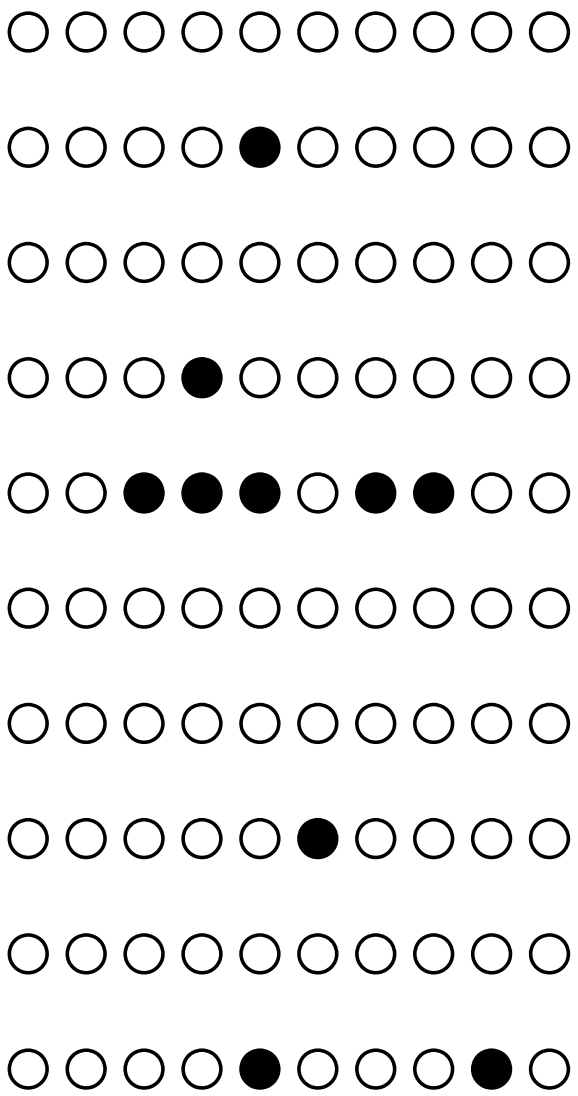
If the numbers of marginal results exceed *c*, the lot is to be rejected. If c=0 (m=M) then it is a two-class sampling plan

How can we be sure to find…?



In how many ways can we select one ● if we sample 5 items?





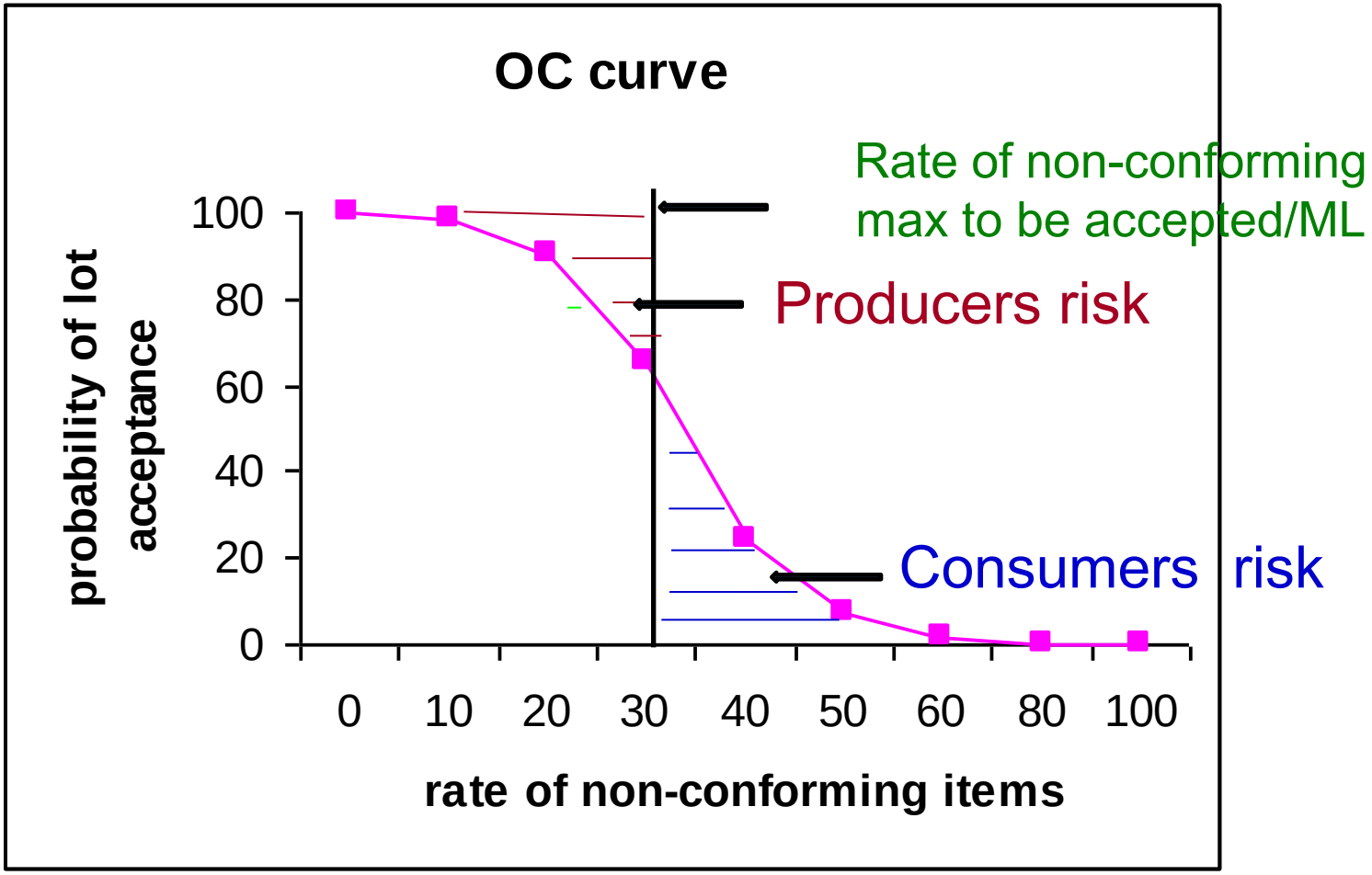
n = 5, P(●○○○○)= P(X=1)?

p(●) = 0,1

What is the Probability **P_A** of accepting a lot when **n** samples are taken and with an acceptance number of **c** if the defective rate is **p**?

$$P_A = P(X = 1) = \binom{n}{c} p^c (1-p)^{(n-c)}$$

OC (Operating Characteristic) – curve



Attribute sampling plans

% Probability of accepting a lot (=P_A), given a % defective rate p

Acceptable Quality Level - AQL = 6,5

% defect ↓	n=2, c=0	n=8, c=1	n=13, c=2	n=20, c=3	n=32, c=5	n=50, c=7
5	90	94	98	98	99	100
10	81	81	87	87	87	91
20 →	64	50	50	41	36	19
30	49	26	20	11	5,1	0,7
40	36	11	5,8	1,6	0,3	0
50	25	3,5	1,1	0,1	0	0

Ref: NMKL Guide no 12, Codex: CAC GL 50 (2004) and ISO 2859-1 (1999)

% Probability of accepting a lot (=P_A), given a % defective rate p for a given sampling plan (n, c). AQL values from ISO 2829-1 1999 for given n and c

Common values for n and c from

EC regulation 2073/2005.

n →	**5**	**5**	**5**	**9**	**10**	**30**
c →	**0**	**1**	**2**	**2**	**0**	**0**
AQL →	**2,5**	**10**	**15**	**10**	**1,5**	**0,65**
% defect p ↓						
5	77	98	100	99	60	21
10	59	92	99	95	35	4
20	33	74	94	74	11	0
30	17	53	84	46	3	0
40	8	34	68	23	1	0
50	3	19	50	9	0	0

Model for determining acceptable/ not acceptable – attribute sampling plans

How many samples **N** must we collect to show that a lot (with undefined number of items) with a probability **P** do not contain more than **p** % defective?

Calculation of the number of items to be collected to attain a given probability of conformance

- *Given*
- P = probability of demonstrating non-conformance
- p = share of non-conforming items in the lot to be sampled
- N = number of samples to be taken from an undefined amount

$$N = \frac{\ln(1-P)}{\ln(1-p)}$$

Calculation of the number of items to be collected to attain a given probability of conformance (2)

- *Ex:* We want to give documentation that eggs (total amount N_0 - restricted in time and place) with a probability of 99 % (P=0,99) do not contain more than 0,1 % (p= 0,001) eggs with *Salmonella*, N eggs must be collected

$$N = \frac{\ln(1-P)}{\ln(1-p)} = \frac{\ln(1-0{,}99)}{\ln(1-0{,}001)} = \frac{\ln(0{,}01)}{\ln(0{,}999)} = 4603$$

If $N > N_0/10$ then N can be reduced according to formula given in NMKL Guide No 12

Example: 25 years ago Red mouth disease (*Yersinia ruckeri)* appeared on Salmon in smolt producing unit in the county of Nordland in Norway

- In the smolt producing units with good conditions the disease could be a latent condition - no symptoms shown on the fish
- Norway had little experience with this disease at the time, but according to literature the prevalence can be about 2 % (p=0,02) in producing units (of smolt or fish in net cages) situated in an infected geographic area - without showing any symptoms

How many fish should be sampled to detect Red mouth disease in such a smolt producing unit?

Number of fish to be collected from a smolt producing unit (latent for Red mouth disease) – to show with a probability **P** will not contain more than **p =2** (in %) infected fishes

Probability P	Producing unit containing max p (in %) infected fish					
(%)	**p = 0,1**	**p = 1**	**p = 2**	**p = 5**	**p = 10**	**p = 15**
99	4 603	458	**228**	90	44	28
95	2 994	298	**148**	58	28	18
85	1 896	189	94	37	18	12
80	1 609	160	80	31	15	10
75	1 386	138	69	27	13	9
70	1 203	120	60	23	11	7
65	1 049	104	52	20	10	6
60	916	91	45	18	9	6
55	798	79	40	16	8	5
50	693	69	34	14	7	4

Example: Red mouth disease on salmon - observation with sampling and analyses

- The local food safety office in Bodø tested several smolt producing units with few or no symptoms. But as they suspected that the units was infected they decided to take more samples and perform analyses. They experienced that to be able to show that at least 1 fish was infected in a smolt production unit they had to analyse at least 150 fish
- If they collected less than 150 fish it was only by chance they could show that the smolt producing unit was infected, although they suspected that was the case

Compliance between theory and practice!

Take an eagles overview before sampling

Thank you for your attention!!