ASTA 2007 Annual Meeting & 100th Anniversary Celebration

Dr. Katrin Hoenicke

Pesticides and Prohibited Dyes in Chili Peppers and Oleoresins

April 16 – 19, 2007

Ritz-Carlton Laguna Niguel
Dana Point, California

Overview

- Pesticides in chili pepper and oleoresins
  - Legislation
  - Occurrence
  - Strategy for testing
- Prohibited dyes in chili pepper and oleoresins
  - Legislation
  - Azodyes (Sudan dyes)
  - Other synthetic dyes
  - Bixin
  - Conclusion and risk assessment

Pesticides Legislation

- International: Codex Alimentarius
- US: EPA tolerances
- Europe:
  - Council Directive 90/642/EEC
  - Regulation (EC) No 396/2005 (new framework regulation, not yet fully implemented)
- National:
  - e.g. Germany: Rückstands Höchstmengen-verordnung

Pesticides legislation
Codex Alimentarius

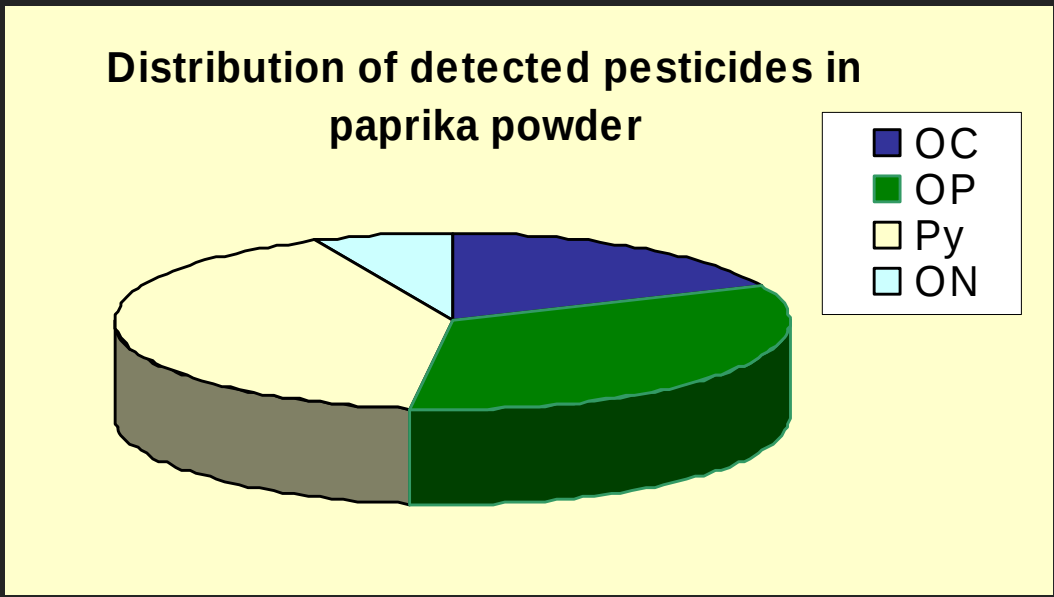
- Sets maximum residue limits (MRLs) for special pesticides in different agricultural commodities
- MRLs are not defined for every active compound
- A concentration factor for processed products (i.e. oleoresins) is not considered

Pesticides legislation
EU legislation

- Sets MRLs for special pesticides in different agricultural commodities
- Usually EU MRLs are lower than Codex MRLs
- For those products not covered by a specific MRL or for active substances not listed in the regulation a general MRL of 0.01 ppm is in force
- MRLs are also in force for processed food
  - Specific concentration or dilution factors for processed products are taken into account

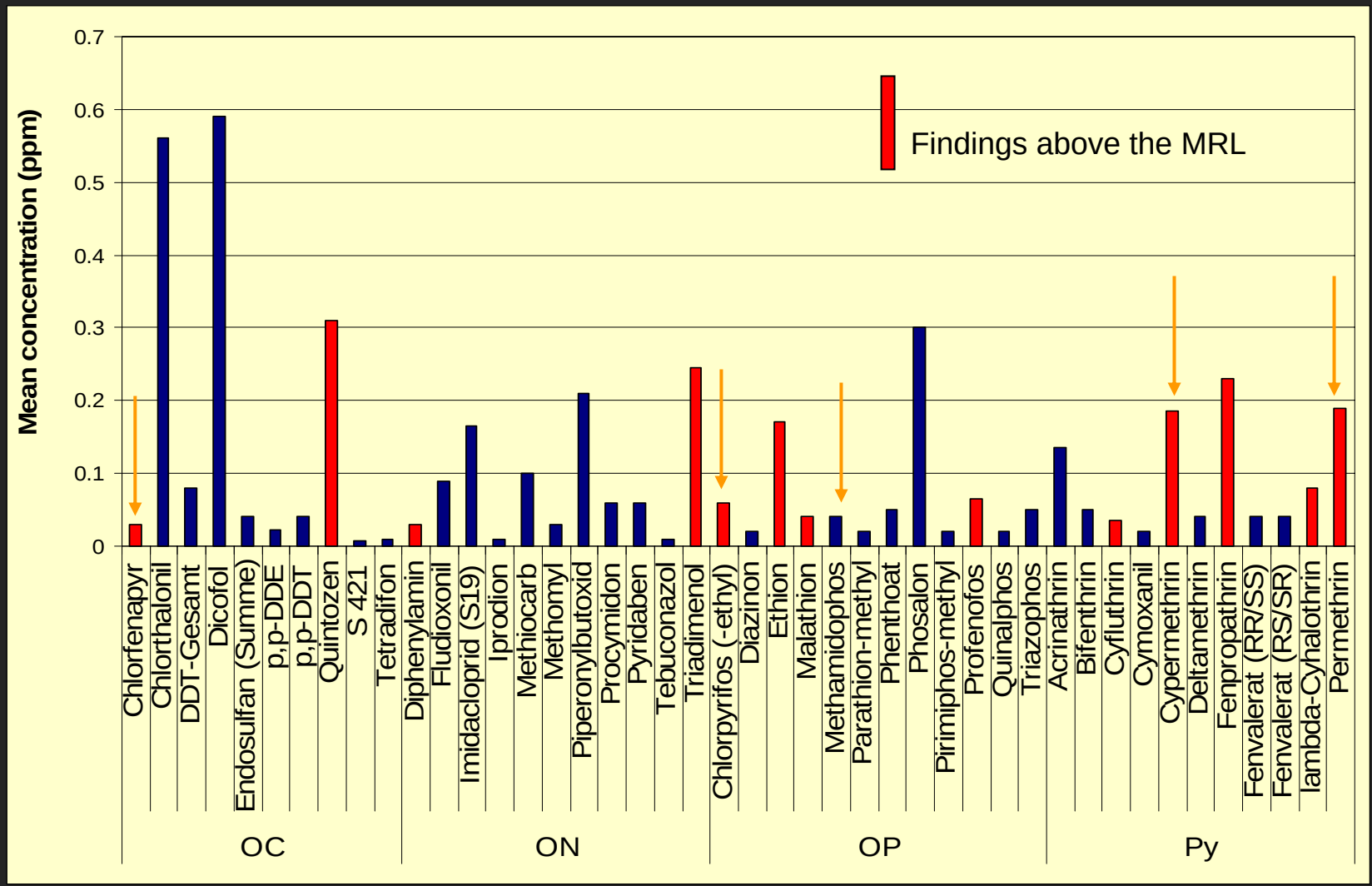
Pesticides results (2006)
Paprika powder

- Mainly pesticides from the group OP and Py were detected



OC: organochlorine pesticides
OP: organophosphorus pesticides
Py: Pyrethroides
ON: organonitrogen pesticides

Pesticides results (2006)
Paprika powder



Pesticides results (2006) Oleoresins

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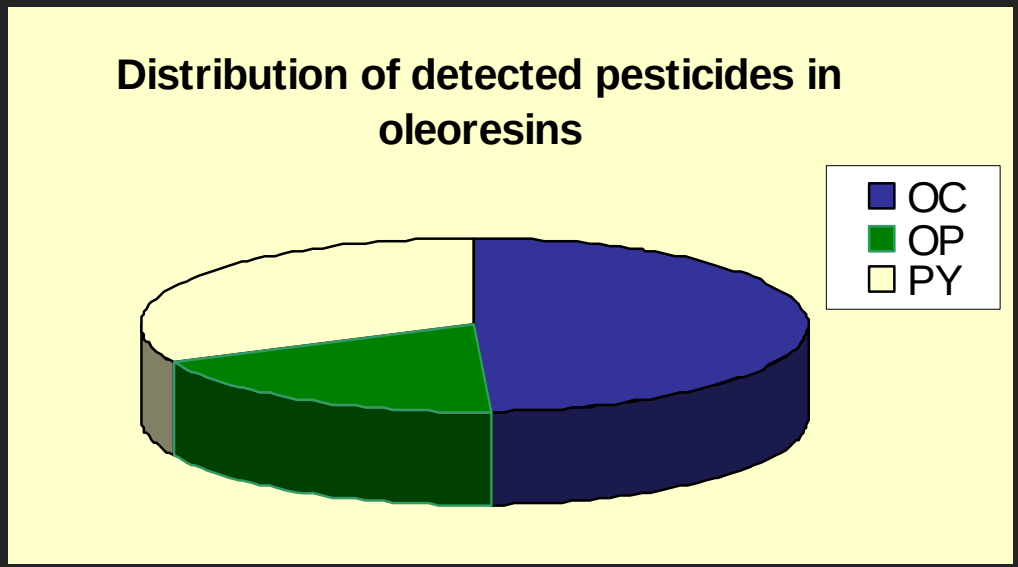

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Pesticides results (2006)
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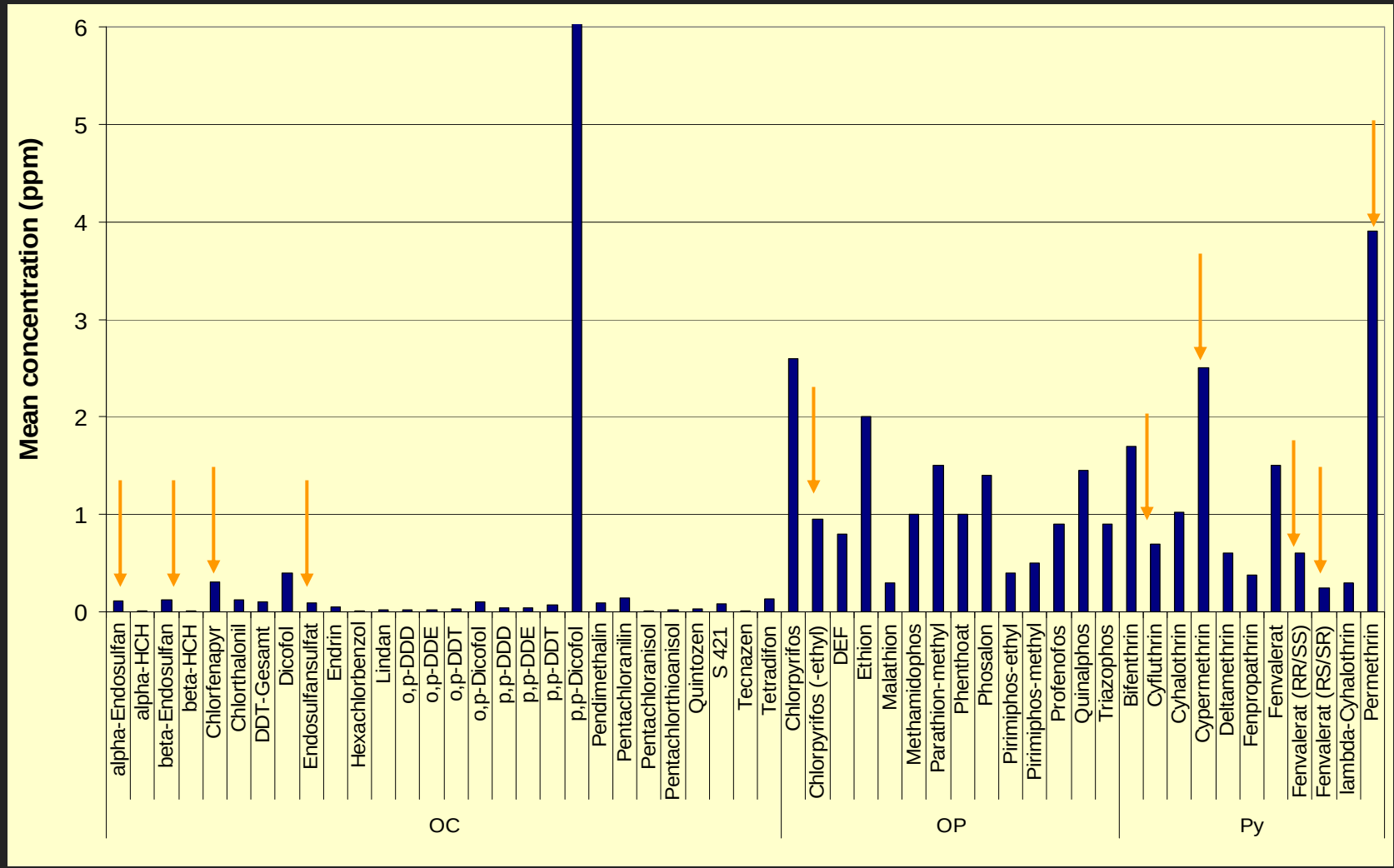
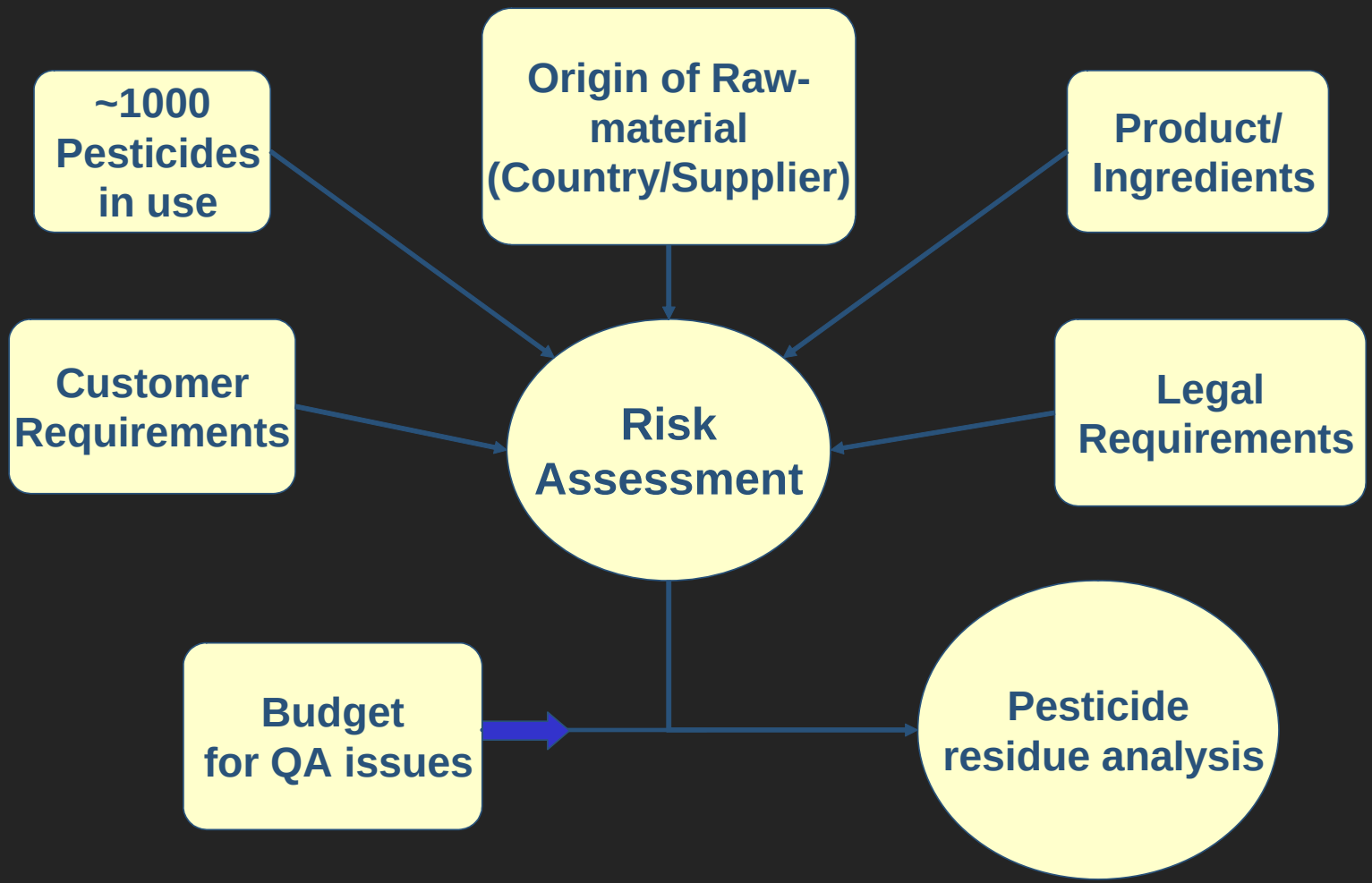


Pesticides
Strategy for testing

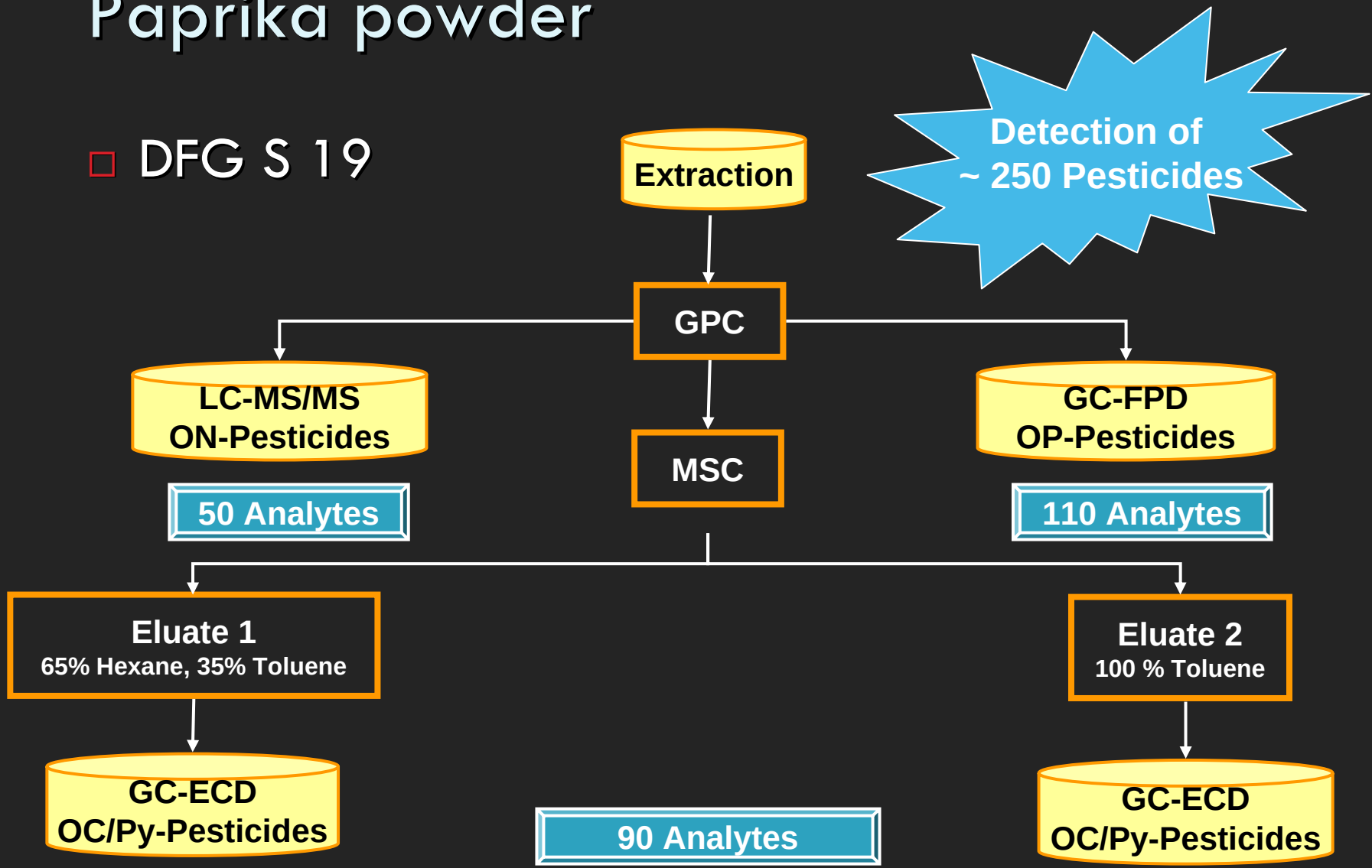


Pesticides
Testing methods

- Must cover a broad spectrum of analytes (multiresidue methods)
- Rugged
- Produce reliable and reproducible results
- Acceptable turn around times
- Acceptable costs considering the requirements mentioned above

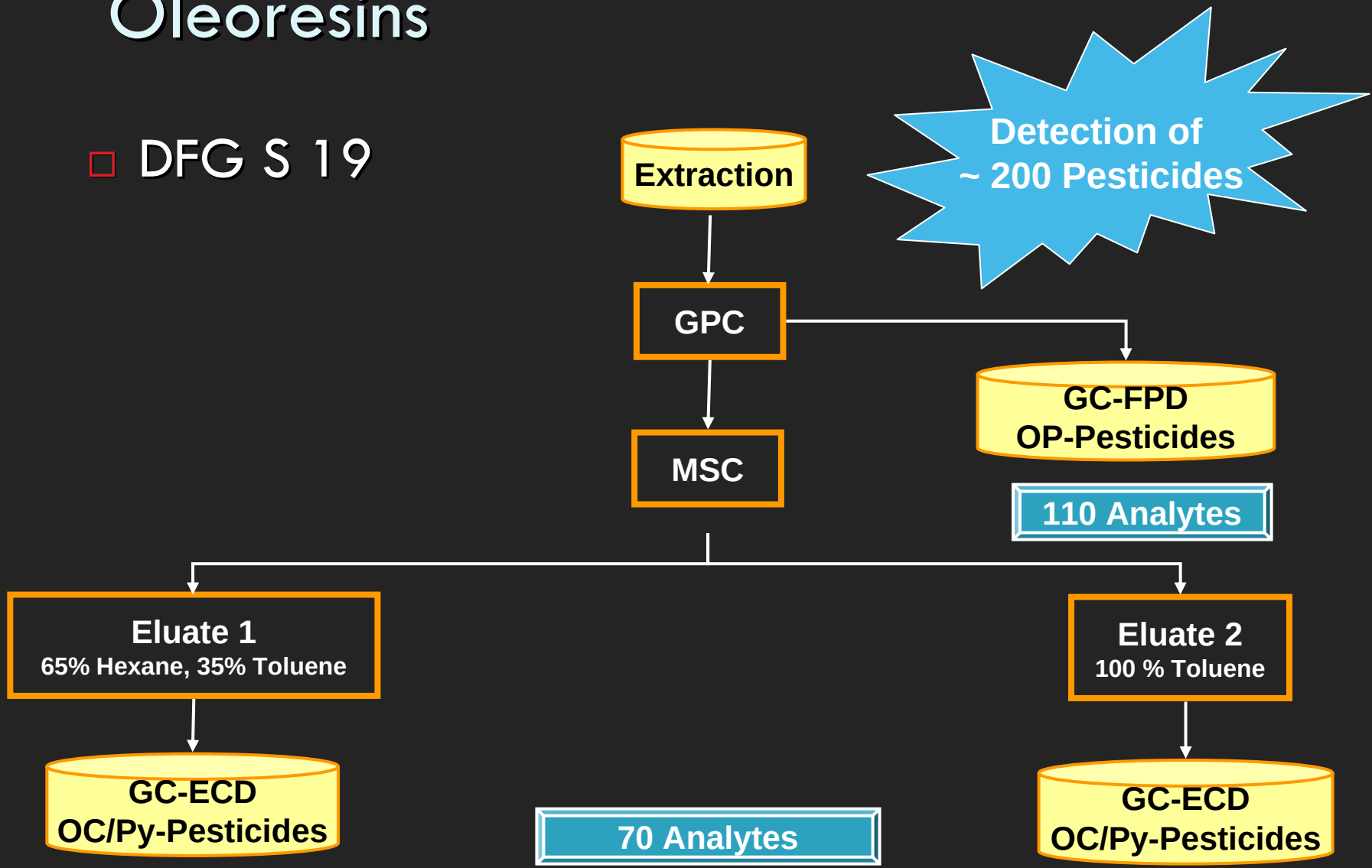
Pesticides testing methods
Paprika powder

- DFG S 19



Pesticides testing methods Oleoresins

- DFG S 19



Adulterants – Prohibited dyes

Aldulterants
Prohibited Dyes

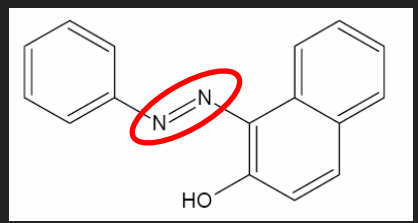
- In May 2003 the European Authority reported the finding of 4000 ppm Sudan I in ground capsicums from India

  -> The use of dyes to manipulate the quality of spices became an issue!

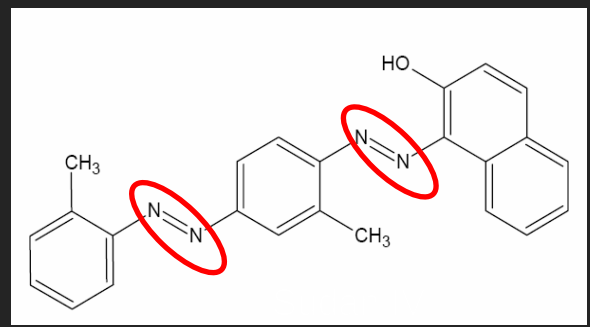
  - Dyes are added to improve the visual (surface) color of capsicum products

Azodyes

- Synthetic dyes
- General structure: $R_1–N=N–R_2$
- Not authorized as food colors in the US / EU
  (degradation products are considered to be carcinogenic and teratogenic (IARC, 1975/1978: Group 3))
- Most important as adulterant in spices:
  Sudan dyes



Sudan I



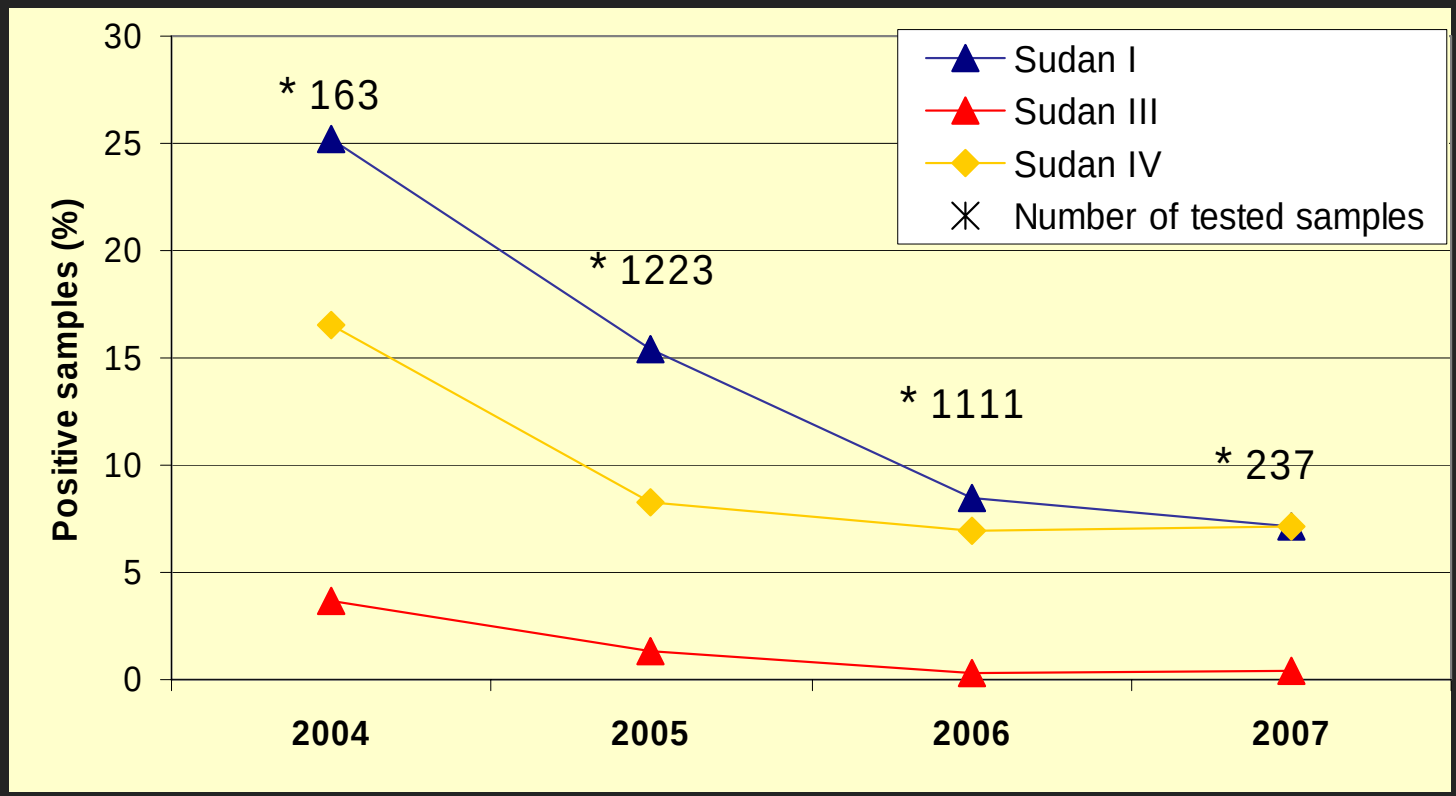
Sudan IV

Azodyes Regulation

- So far there is zero tolerance for these dyes (not part of the list of permitted food colours, Directive 94/36/EC)
- EU:
  - Decision 2003/460/EC: Requires as a condition for importation that all hot chili and hot chili products have to be tested for Sudan I
  - Decision 2004/92/EC: Includes additional testing for Sudan II, III and IV
  - Decision 2005/402/EC: Includes a requirement for additional raw materials to be tested (turmeric and palm oil)

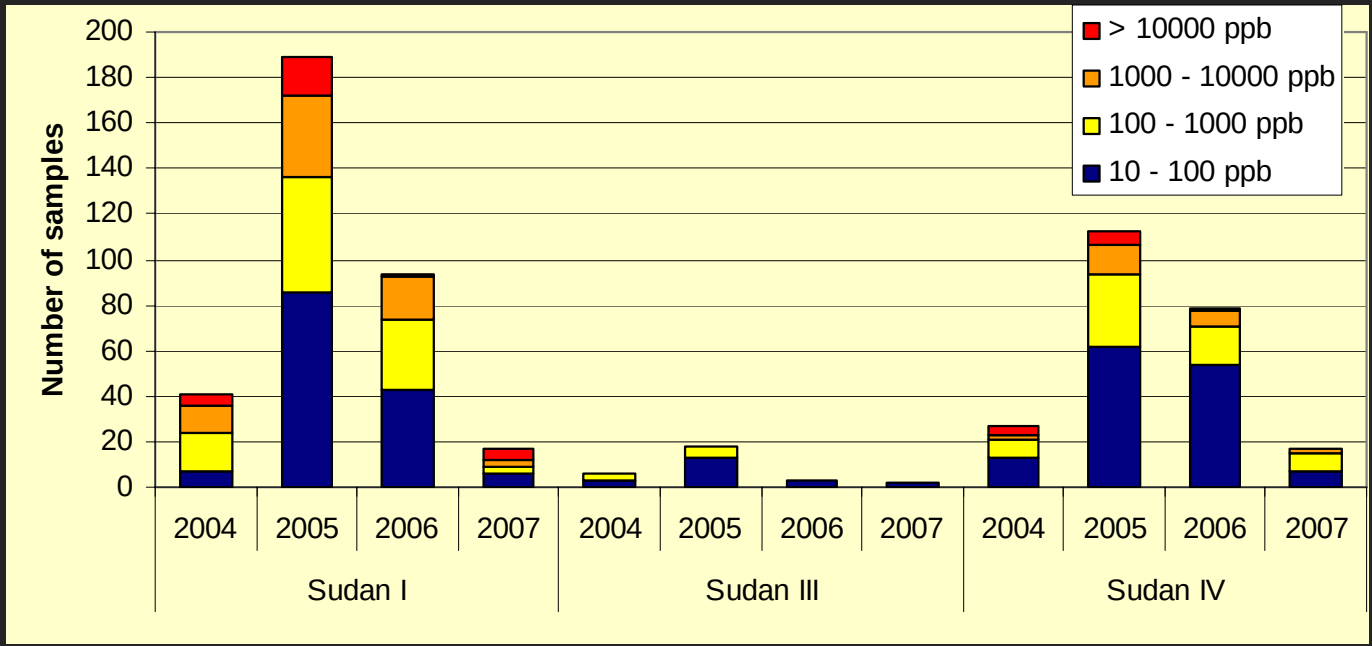
Azodyes Results
Sudan dyes in chili pepper

- Detected: Sudan I, Sudan III and Sudan IV



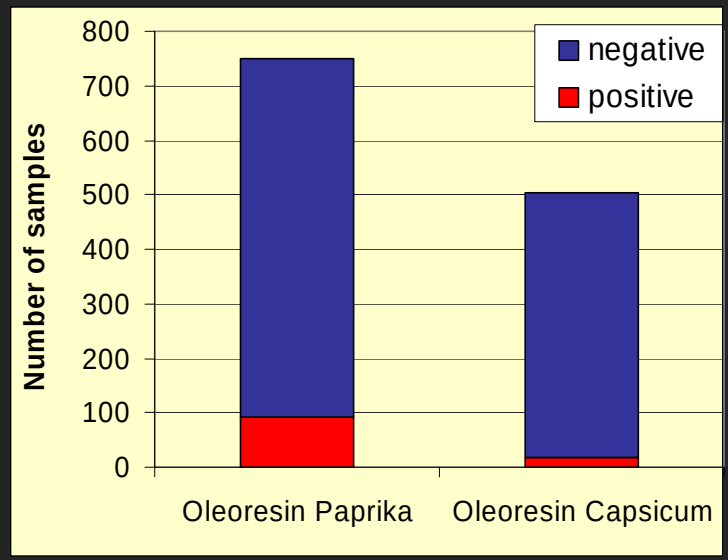
Azodyes Results
Sudan dyes in chili pepper

Number of positive samples and determined values



Azodyes Results
Sudan dyes in oleoresins

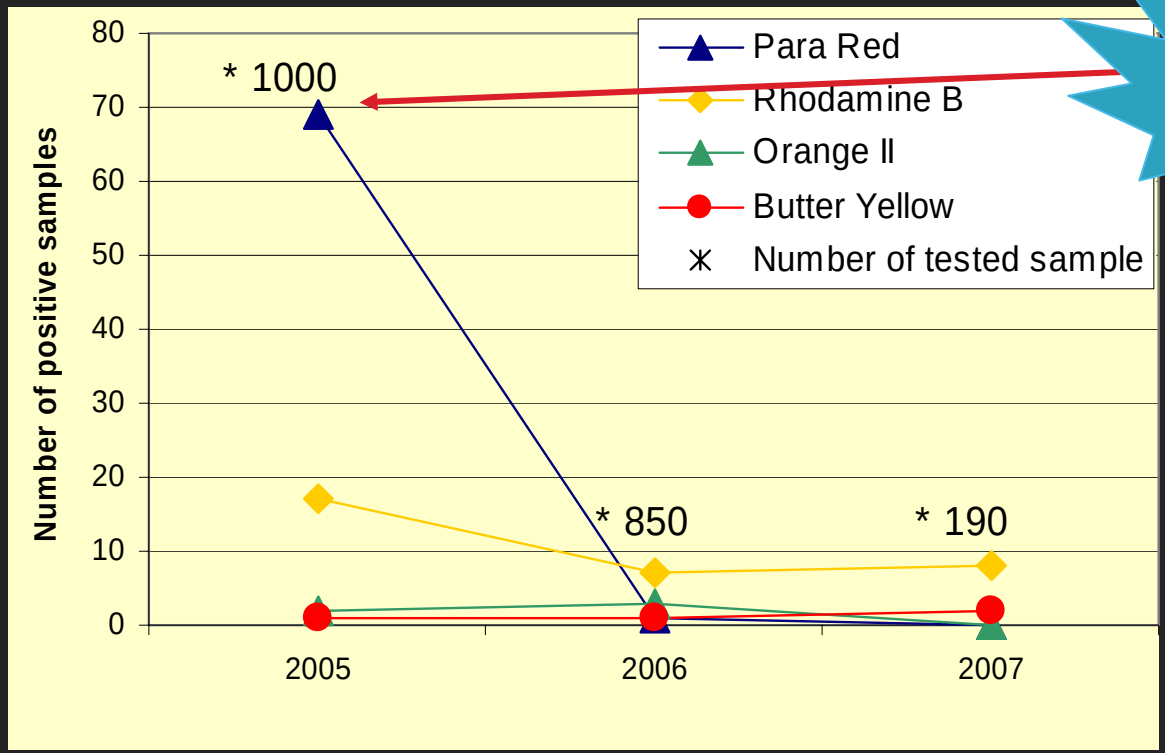
- Detected: Sudan I
- Determined values:
  only 10 – 200 ppb!!



- Cause: Lubricants containing Sudan I are used for greasing of the extraction plant!!!

Other synthetic dyes
Chili pepper

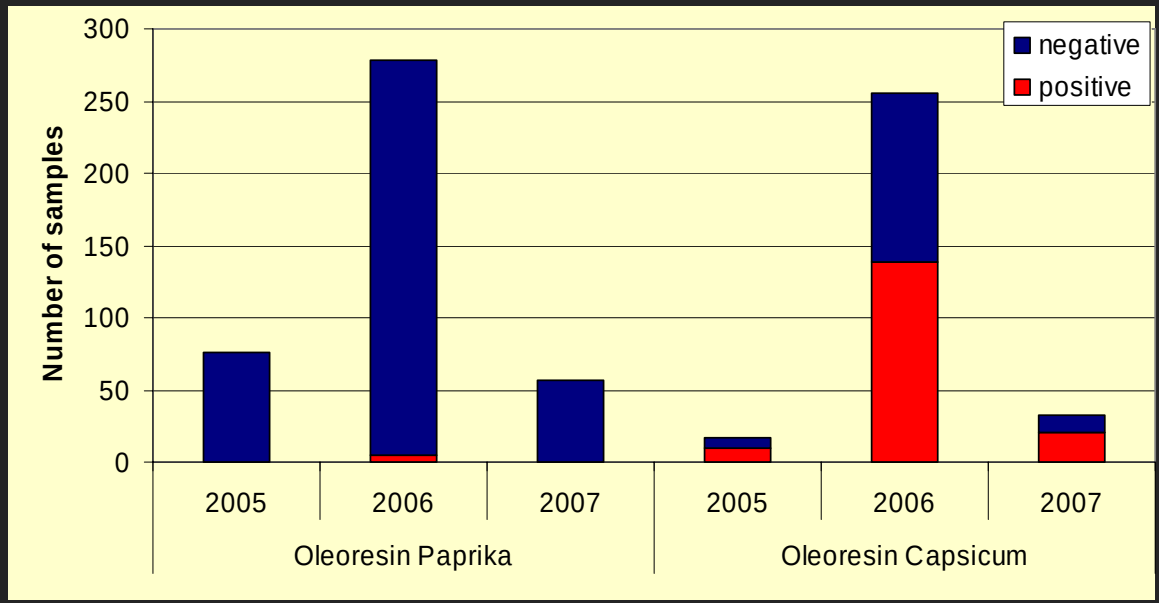
- Other synthetic dyes found so far:
  - Para Red, Rhodamine B, Orange II, Butter Yellow



Para Red incident in UK

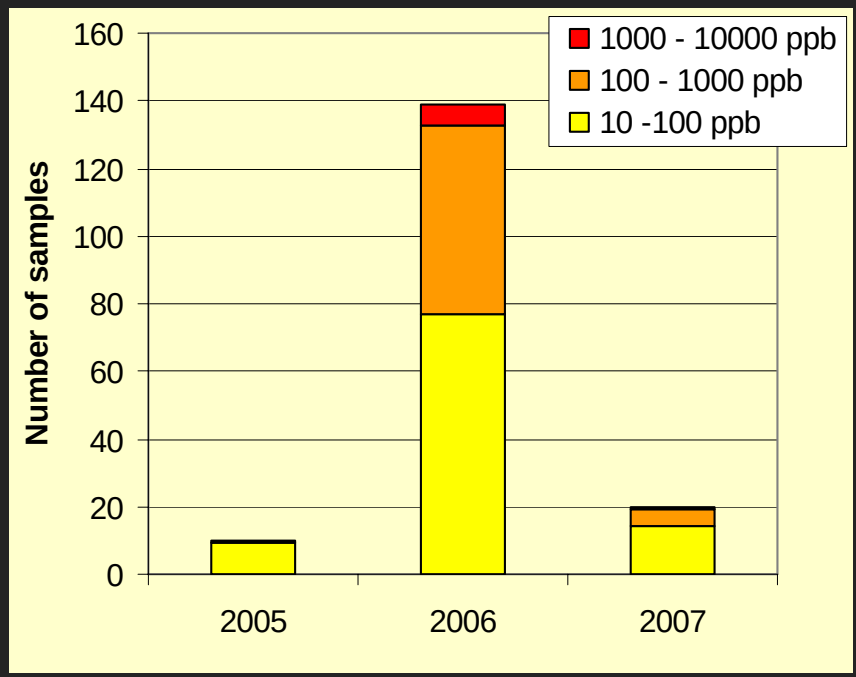
Other synthetic dyes
Rhodamine B in oleoresins

- Rhodamine B was detected especially in capsicum oleoresin
  - Here, 50% of the analyzed samples tested positive!!!



Other synthetic dyes
Rhodamine B in oleoresins

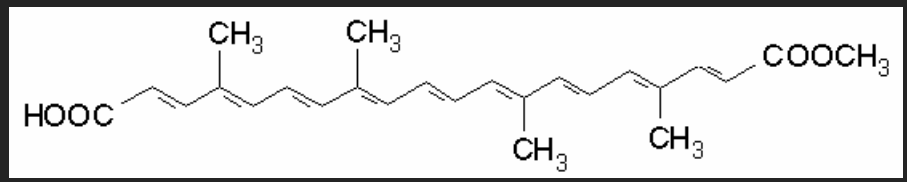
Number of capsicum oleoresin samples tested positive and determined values



Cause???

Other adulterants
Bixin

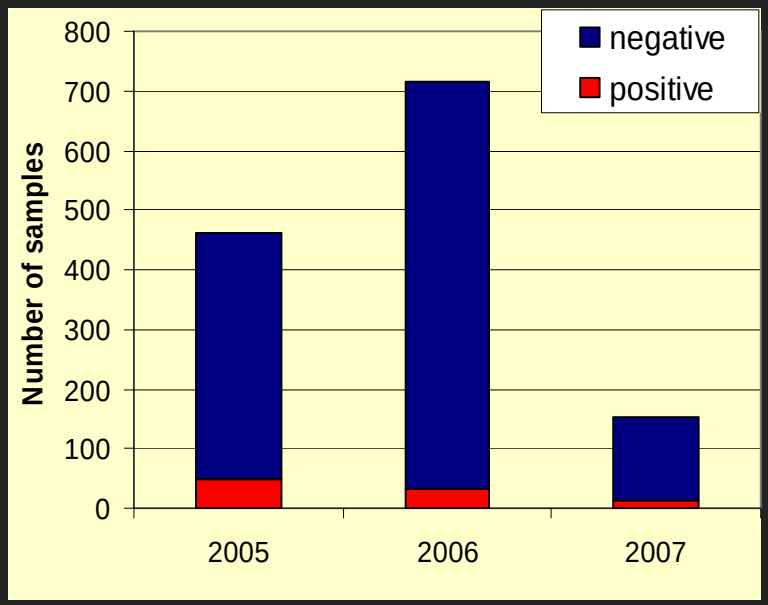
- The carotinoid bixin is the main component of the natural food colorant Annatto (E160b)

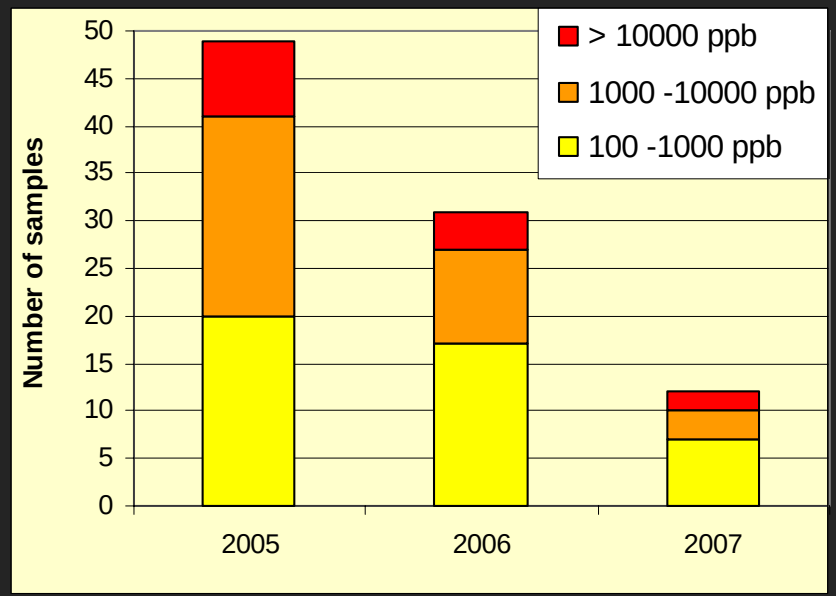


- Annatto is permitted for use in a variety of foods but *not* in spices

Other adulterants
Bixin in chili pepper

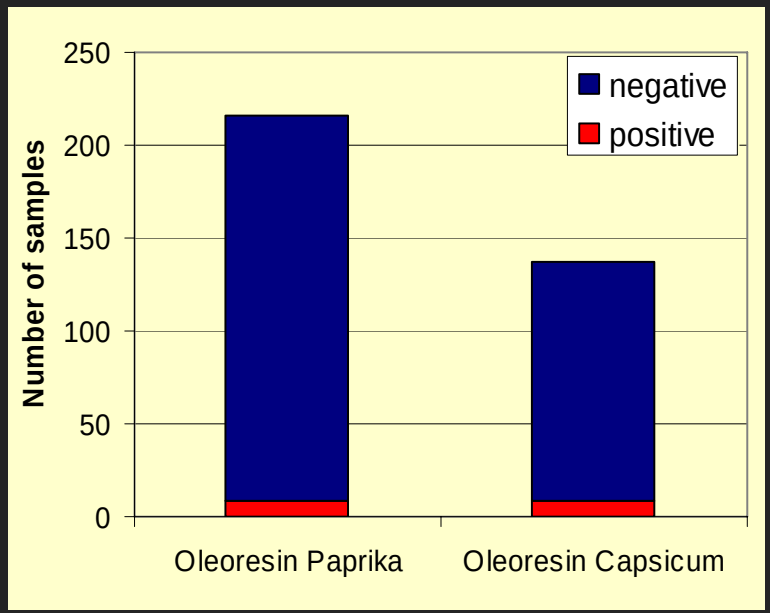
- Bixin could be detected in 5 - 10 % of the analyzed samples
- Determined values: 100 µg/kg – 1.6 g/kg

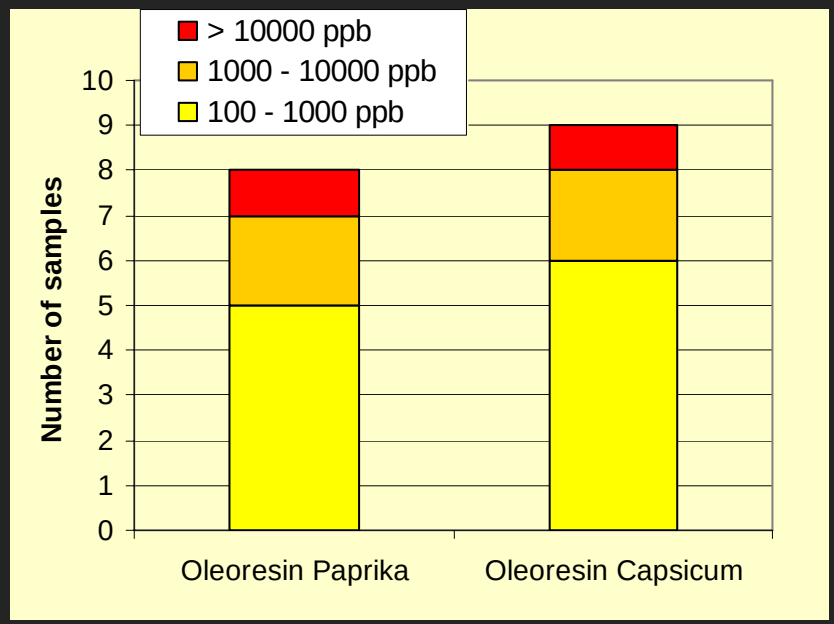




Other adulterants
Bixin in oleoresins

- Bixin could be detected in ~ 5 % of the analyzed samples
- Determined values: 100 µg/kg - 100 mg/kg





Prohibited Dyes Conclusion

- Chili Pepper:
  - Most important: Sudan I-IV
  - Also important: Bixin, Rhodamine B, Para Red, Butter Yellow, Orange II
- Oleoresin:
  - Most important: Rhodamine B, especially in capsicum oleoresin
  - Also important: Sudan I-IV, Bixin

Prohibited Dyes Recommendations

- **ESA** (European Spice Association):

  All incoming materials should be checked for:

  - Sudan I–IV, Sudan Red 7B, Sudan Red G, Sudan Orange G, Sudan Red B
  - Para Red, Butter Yellow, Orange II, Rhodamine B
  - Bixin
  - Toluidine Red, Metanil Yellow

Prohibited Dyes Recommendations

- EFSA (European Food Safety Authority):

  Dyes used illegally in countries from which spices originate and viewed as genotoxic and/or carcinogenic
  - Sudan I-IV, Sudan Red 7B, Sudan Red G
  - Para Red, Butter Yellow, Orange II, Rhodamine B
  - Malachite Green, Leucomalachite Green
  - Ponceau 3R, Ponceau MX, Acid Red 73, Congo Red, Naphthol Yellow
  - Orange OT (= Oil Orange SS), Metanil Yellow, Auramine

Risk assesment

Elements which should be considered as part of a risk assessment*:

- Country of origin
- Nature of the material (whole, ground, etc.)
- Type of spice
- Supplier selection and approval:
  - Raw material control
  - Capability of meeting legislative requirements
  - Adherence to Good Manufacturing Practice (GMP)
  - Adherence to HACCP principles
  - Traceability
  - Third party certification
  - Testing capabilities and accreditation to ISO 17025

* Source: ESA Advice, June 2006

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Thank you!

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