

Update on FAPAS acrylamide series

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FAPAS

Food

Analysis

Performance

Assessment

Scheme

Established in 1990, covers a wide range of test materials/analytes, worldwide

FAPAS proficiency materials to date

SERIES 30. Acrylamide

R1. Crispbread

R2. Potato crisps (chips)

R3. Breakfast cereal

R4. Coffee

R5. Crispbread

R6. Breakfast cereal

R7. Oven chips (French fries) (sent out March-04)

FAPAS - acrylamide

- Test materials are 'naturally incurred'
- Materials are tested for sufficient homogeneity
- Results are examined to find the assigned value which is the best estimate of the true concentration and is a consensus value

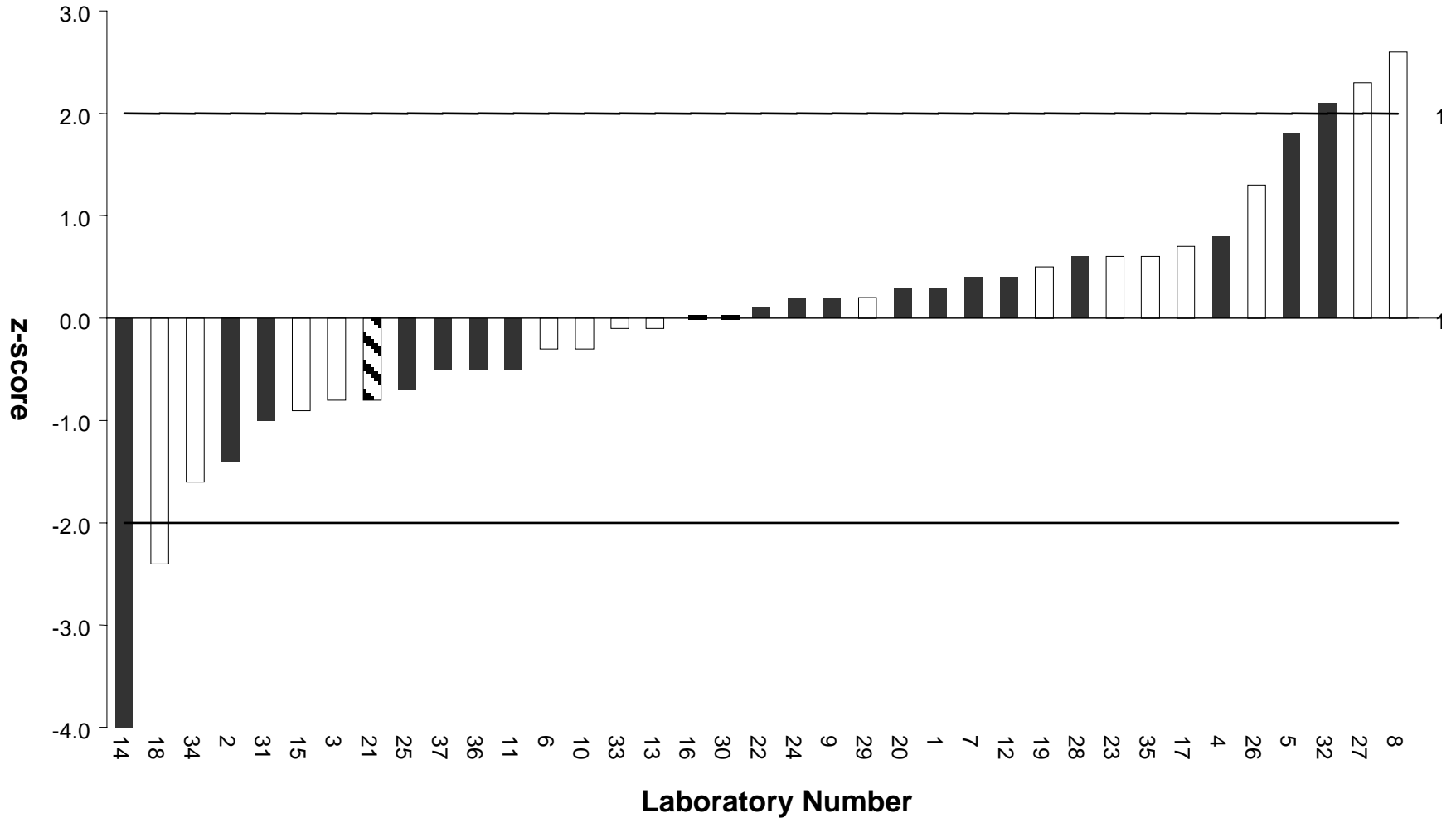
Boundaries for:

- Satisfactory $-2 < z < 2$
- Questionable $-3 < z < 3$
- Not satisfactory z outside ± 3

Performance set empirically using Horwitz/modified Horwitz equation

General format for FAPAS z-scores

Acrylamide Round 1. Crispbread. Sept-02



FAPAS Test materials

| Round Date | R1 Jul-02 | R2 Nov-02 | R3 Feb-03 | R4 Jul-03 | R5 Aug-03 | R6 Nov-03 |
|-----------------------------|-----------------|-----------------------------|--------------|--------------|-----------------|--------------|
| Test material | Crisp- bread | Potato crisps (chips) | Cereal | Coffee | Crisp- bread | Cereal |
| Assigned value (ppb) | 1213 | 167 | 109 | 312 | 707 | 95 |
| Satisfactory range (ppb) | 836- 1590 | 97-237 | 61-158 | 193- 431 | 468- 945 | 53-137 |

Performance in acrylamide analysis

| | R1 | R2 | R3 | R4 | R5 | R6 |
|----------------|-----|-----|-----|-----|-----|-----|
| Return rate | 67% | 95% | 85% | 76% | 79% | 64% |
| Participants | 37 | 40 | 35 | 31 | 45 | 29 |
| % satisfactory | 86% | 87% | 71% | 87% | 71% | 82% |

Accredited labs - % satisfactory

| | R1 | R2 | R3 | R4 | R5 | R6 |
|------------------------------|--------|---------|---------|--------|---------|--------|
| % lab accredited | 7 / 37 | 11 / 40 | 10 / 35 | 9 / 31 | 12 / 45 | 9 / 29 |
| % satis. by these labs | 100% | 91% | 80% | 100% | 66% | 100% |

Measurement technique used

% satisfactory

| | R1 | R2 | R3 | R4 | R5 | R6 |
|-----------|-------------|------------|------------|------------|------------|-------------|
| GC-MS | 48% 84% | 46% 89% | 39% 79% | 35% 91% | 44% 70% | 53% 73% |
| LC-MS(MS) | 52% 91% | 54% 90% | 61% 68% | 65% 85% | 53% 76% | 47% 86% |
| other | ECD 100% | - | - | - | DAD 0% | DAD 100% |

Examination for sign of bias

average z-score (satisfactory scores only)

| | R1 | R2 | R3 | R4 | R5 | R6 |
|----|-------|-------|-------|-------|--------|-------|
| GC | -0.09 | -0.11 | 0.1 | 0.15 | 0.41 | -0.07 |
| LC | 0.04 | -0.24 | -0.03 | -0.14 | -0.006 | -0.21 |

Extraction solvent used

| | R1 | R2 | R3 | R4 | R5 | R6 |
|---------|-----|-----|-----|-----|-----|-----|
| Water | 62% | 82% | 77% | 77% | 70% | 80% |
| Aqueous | 16% | 5% | 6% | 16% | 19% | 13% |
| Non-aq. | 22% | 13% | 17% | 6% | 11% | 7% |

Non-aqueous z-scores by round

| | R1 | R2 | R3 | R4 | R5 | R6 |
|----------|------------|------|---------------------|-----|----------|------|
| MeOH | -0.3, -4.0 | -0.3 | 49.9 | | | -2.1 |
| PrOH | 0.5, 1.3 | 11.2 | 0.2 | 2.3 | 1.4, 0.8 | |
| EtOAc | 2.6 | -0.8 | 15.6, 1.6, -0.4, | 0.0 | | |
| DCM/EtOH | 2.1 | -3.1 | -1.9 | | | |
| DCM | | | | | -4.5 | |
| MeCN | | | | | -0.5 | |

Round 30-06

Direct (underivatized) GC analysis

n=4 labs

Z= 2.7, 0.2, 2.6, 1.4

Extraction = HW, CW+EtOAc, HW, CW+PrOH

FAPAS proficiency materials - planned

SERIES 30. Acrylamide

R7. Oven chips (French fries) (sent out March-04)

R8. Coffee July-04

R9. Crispbread Sept-04

R10. Breakfast cereal Nov-04

R11. Potato crisps (chips) March-05

FAPAS Acrylamide rounds conclusions

- Plain water extraction seems OK
- GC & LC methods seem to be equivalent
- Underivatised GC method may be problematic ?
- Accuracy seems OK
- Is the precision really acceptable ?

FAPAS - acrylamide

(end)