Report By Members of Group C: The Wide or Survey Scan

Group Members:

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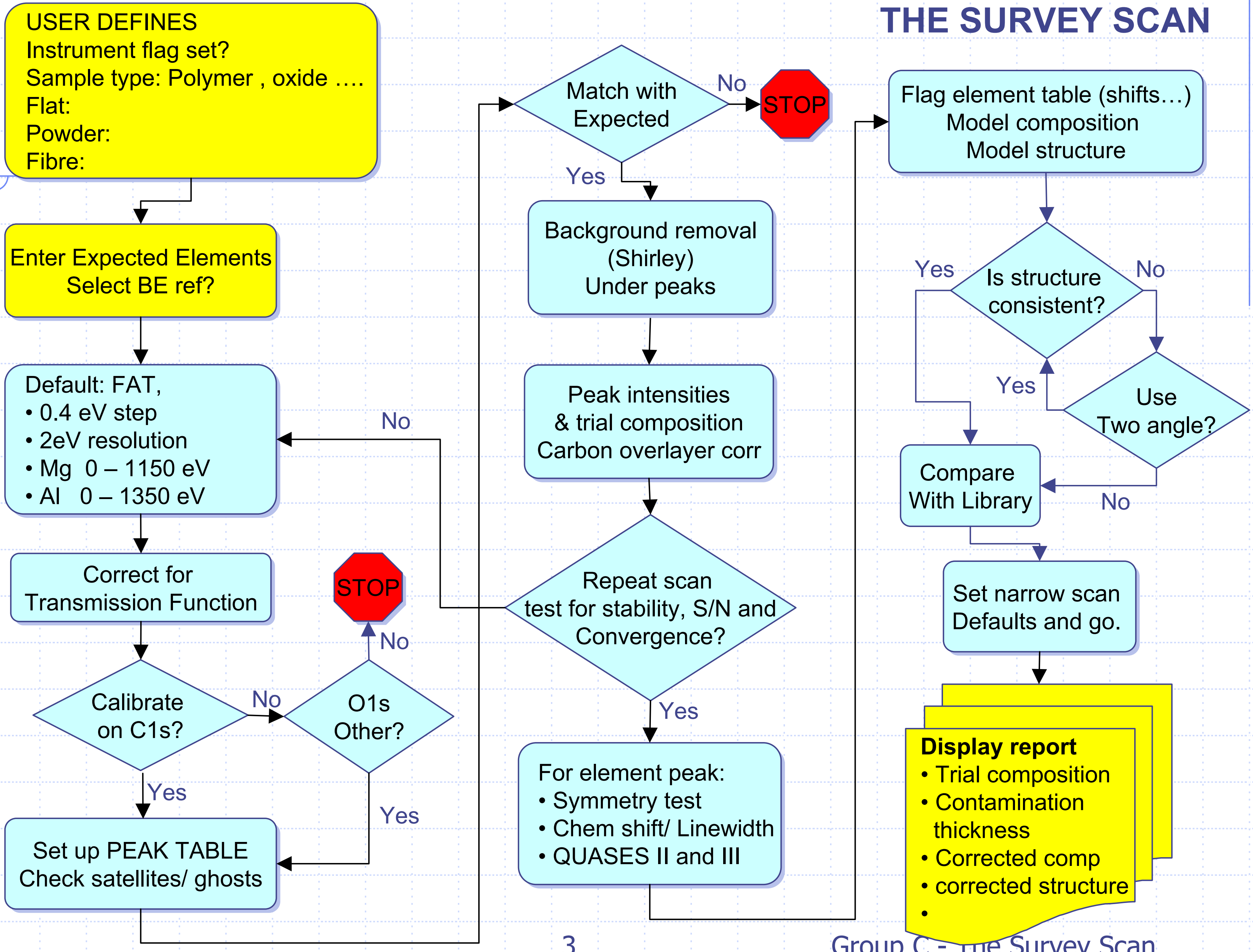
Mission: ‘Qualitative Assessment of Quantitative Opportunity’

Task: To produce an implementable protocol for the collection and analysis of a standard survey scan; allowing extraction of the maximum available information. This information *may* then guide the next steps in sample analysis.

Output: A flow chart illustrating essential steps in the acquisition of a survey scan, and especially inferencing points where rules would be needed. Initial development of the underlying rule set that guides the decision tree.

THE SURVEY SCAN



USER DEFINES
Instrument flag set?
Sample type: Polymer , oxide ….
Flat:
Powder:
Fibre:

Enter Expected Elements
Select BE ref?

Default: FAT,
- 0.4 eV step
- 2eV resolution
- Mg 0 – 1150 eV
- Al 0 – 1350 eV

Correct for Transmission Function

Calibrate on C1s?

Yes

Set up PEAK TABLE
Check satellites/ ghosts

No

O1s Other?

No

STOP

Yes

Match with Expected

No

STOP

Yes

Background removal
(Shirley)
Under peaks

Peak intensities
& trial composition
Carbon overlayer corr

Repeat scan
test for stability, S/N and Convergence?

No

Yes

For element peak:
- Symmetry test
- Chem shift/ Linewidth
- QUASES II and III

Flag element table (shifts…)
Model composition
Model structure

Is structure consistent?

Yes

No

Use Two angle?

Yes

No

Compare With Library

Set narrow scan
Defaults and go.

Display report
- Trial composition
- Contamination thickness
- Corrected comp
- corrected structure
-

Topics Considered

- Instrument Condition
- Sample Description
- Resolution
- Range
- Charge Correction
- Peak Identification and Labelling
- Peak Intensity
- Element-Specific Data
- Assessment and Utilisation of background
- Assessment and Utilisation of Peak Intensities
- Assessment of whole Spectrum
- Other Options
- Output to Screen
- Region Set-Up

Instrument Condition

The assumption underlying the acquisition is that the instrument is in good health. That is calibrated for linearity; transmission function; X-ray condition and detector efficiency. We assumed that logs were maintained and that events such as open-to-air would be included. **Recommendation:** ***Flag(s) should be set for instrument state and made available during the acquisition sequence and to the output screen compiler if required.***

Sample Description

The ISO format is largely OK but needs augmenting.
Recommendation: ***include air-exposed? Elements sought? Charge reference peak (default is unset)***

Resolution

The main task of the survey scan is to catch all peaks in the most time-efficient manner and to minimise the possibly adverse impact of chemical shift on peak recognition. **Recommendation:** ***FWHM (Ag3d) 2eV***

Range, step size and acquisition mode

Be wide enough to include C_{KLL} Auger and other potentially valuable peaks.
Recommendation: ***1150eV for Mg $K\alpha$ and 1350eV for Al $K\alpha$.***
Have adequate steps to obtain peak area and use FAT to match high resolution scans and permit use of QUASES and other algorithms.
Recommendation: ***0.4eV step size in FAT mode***
Multiple scans permit analysis of drift in peak position and intensity with time. Scans could seek convergence of trial composition.
Recommendation: ***Use repeat scans against suitable convergence or S/N test, ending on maximum number of scans if necessary.***

Charge Correction (Wide scan)

First default is C1s from adventitious carbon assessed using suitable set of inference rules. Those published need augmenting to be fail-safe and additional rules would be useful. For example:

Carbon_1s_intensity/Carbon_KLL_intensity < (ball_park_figure)

The second default is oxygen 1s.
Recommendation: ***Develop rules for O1s***

These defaults would be implemented if, for example:

Sample_environment_exposed is *yes*
and
Specified_charge_reference_peak is *no*

Recommendation: ***Correct whole spectrum for charge shift using inference engine and record magnitude of shift***

Peak Identification and Labelling

Peak identification is more important than current advisory use. It forms the basis for all real-time data processing.
Recommendation: ***All peaks should be labelled: a) make increased use of pattern recognition, b) include positions of any elements to be sought, c) note positions of CuLα, OKα and Mg/Al cross-over ghosts.***

Peak Intensity

The Shirley background should be used but we noted that the start positions are element specific. A look-up table is necessary. The Shirley should revert to linear background for negative background step.

Recommendation: ***Note essential need for look-up table for energy range and other forms of processing. Recommend development of standard format for look-up tables.***

Element-Specific Data

Recommendation: ***Include peak positions and cross-sections, EAL parameters, satellites, background characteristics, range of chemical shifts, overlaps, useful Auger peaks, plasmon positions.***

Assessment and Utilisation of Peak Intensities

Produce table of Atomic % composition, based on homogeneous model.
Assess overlayer thickness based on carbon intensity.
Assess overlayer thickness based on peak ratios of main constituents.
Seek convergence if necessary, using O1s.
Recommendation: ***Develop inferencing rules for structural assessment***

Assessment and Utilisation of Background

Background shape and intensity relative to peak gives information on surface/substrate distribution, composition and direction of concentration gradients.

Recommendation: ***Run QUASES ll and lll (or other suitable simulation software) on major peaks of all elements, where possible (e.g. region clear of peaks for 30eV in BE above the peak).***

Assessment of whole Spectrum

Compare with local and remote spectral libraries.

Recommendation: ***Develop standard library format to assist in search and compare***

Output to Screen

The spectrum plus:

- Health warning if instrument flag indicates an issue
- Evidence of sample charging during acquisition
- Evidence of composition change during acquisition
- Uncorrected composition
- Estimation of equivalent overlayer thickness using a) carbon, b) peak ratio
- Corrected composition (using overlayer consensus)
- Information from background (e.g. direction of concentration gradients)
- Evidence of chemical state from satellites
- Similar Spectra from Database are …..

Next Steps

- ***Consider whole scan angle ratio (as e.g. suggested by Cumpson).***
- ***Change area?***
- ***Move on to high E resolution scans.***

The way forward!

- Manual testing on a variety of samples and spectra.
- Trial implementation of algorithm on existing data.
- Implementation on real instrument.
- Make widely accessible!

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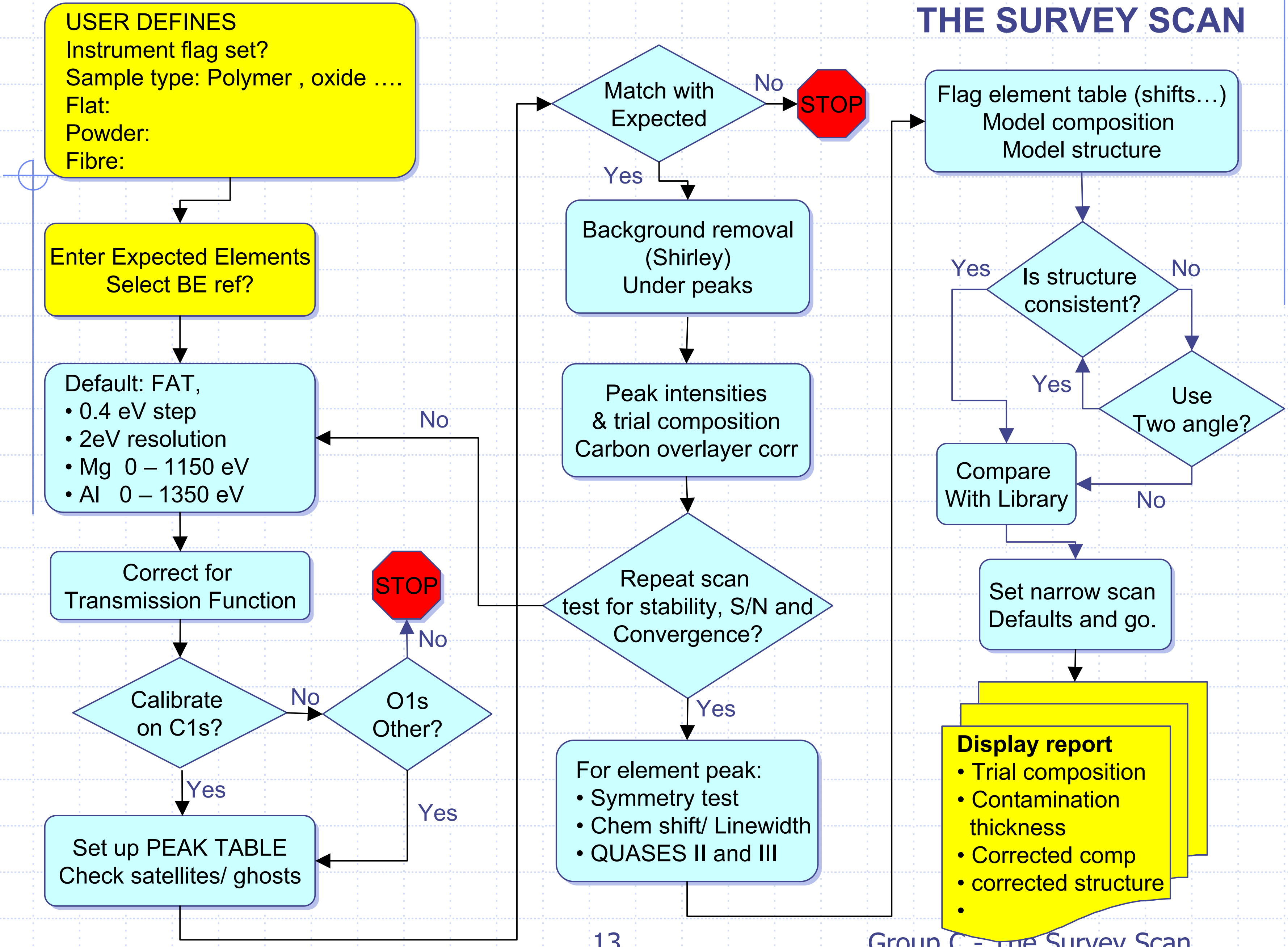

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