P-I236 Analytics (Update 01-02-2010)
From the discussions between the Al salts Small TF Members (11/01/2010 and 19/01/2010) on tests to perform

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As the AlFe REACH Consortium deals with inorganic substances, the analytical tests and especially spectral analyses need to be adapted accordingly. In accordance with the Article 10 and Annex VI of REACH (attachment1) and ECHA Guidance on Substance Identification and Naming, Section 4.2.1.3, the following Conclusions and recommendations are made, to be further adopted by the Technical Committees:

A/ Al salts sub-Consortium

For Identification, key spectral analyses to be performed are

- **Inductively Coupled Plasma spectroscopy (ICP), as it provides the best available method describing sufficiently the spectra**
  OR
- **Atomic Absorption Spectroscopy (AAS), as a supporting spectral method**
  OR
- **X-Ray Fluorescence (XRF). On solid samples X-Ray Diffraction (XRD) is recommended, as a supporting spectral method when the substance is crystalline)**

For quantification of substances, European Standard EN 1302 methods are to be used, such as complexometric titration.

Analysis of each specific element: For determining the amount of water free active components shall be analysed as proposed in Annex 2.

B/ SodAl sub-Consortium

For Identification, key spectral analyses to be performed are

- **Inductively Coupled Plasma (ICP as ICP-OES or ICP-AES) for quantification of multi-elements both in solutions and solids**
  AND
- **Infra-red Spectroscopy (IR) with dry substance (110 °C overnight in oven) with a reference substance**
- **Sodium shall be analysed by Atomic Absorption spectroscopy technique (AAS), unless measured by ICP**

For quantification of substances, European Standard EN 1302 methods are to be used.

C/ Fe salts sub-Consortium

For Identification, key spectral analyses to be performed are

- ***Inductively Coupled Plasma (ICP)***
  AND
- ***Infra-red spectroscopy (IR) only for ferric sulfate and ferrous sulfate***
  OR
- **X-Ray Fluorescence (XRF). On solid samples X-Ray Diffraction (XRD) is recommended, as a supporting spectral method when the substance is crystalline)**

For quantification of substances, European Standard EN 1302 methods are to be used.

- Elemental analysis of all elements (not just the metal) is essential.
- Water content might be useful since hydrogen content cannot be done easily

D/ Justifications for the methods not recommended:

Nuclear Magnetic Resonance (NMR) spectra are not recommended for any of these substances, as they are scientifically not necessary to support the above mentioned analyses. Additionally, NMR is technically not reliable for aluminium as only one bandwidth is available. NMR shows tetrahydral aluminium which is only in Aluminium basic solution, which is quite different from Al chloride. For iron, NMR is not possible as it is a magnetic material.

IR-spectra are not recommended for aluminium salts, because it is scientifically not necessary to conduct these analyses for substances where other analytical spectra, such as ICP are available (Annex 2).

Mass spectra are not recommended, as it is technically not possible to carry out mass spectra on concentrated aluminium or iron salt solutions. Mass spectra can only be made on dilute solutions. When aluminium and iron salts are diluted by water a lot of hydrolytic reactions take place which changes the composition of the salts.

UV-spectra are not recommended, as they are scientifically not necessary: UV-spectra are usually not carried out on inorganic salts as they have no specific UV-spectra and give little information on the composition of the salts. UV of aqueous solution is pointless due to instability and precipitation; acid solution could be used but this limits the value.

GC chromatography is not recommended as it is technically not possible to perform gas chromatography of these inorganic substances. HPLC chromatograms or ion chromatography are neither technically not applicable for these substances.

References:
EN 1302:1999 Chemicals used for treatment of water intended for human consumption – Aluminium-based coagulants - Analysis methods. 52 p.

Attachments:

1 IR & XRF spectra.doc by David COMPTON (ICL)

2 P_I236_ALFE_Proposal for Substance Identity_draft 2.doc by Lars GILLBERG (KEMIRA)

Article 10

Information to be submitted for general registration purposes

"*A registration required by Article 6 or by Article 7(1) or (5) shall include all the following information: […] a technical dossier including (ii) the identity of the substance as specified in section 2 of Annex VI;*"

ANNEX VI

INFORMATION REQUIREMENTS REFERRED TO IN ARTICLE 10

"*2. IDENTIFICATION OF THE SUBSTANCE*

For each substance, the information given in this section shall be sufficient to enable each substance to be identified. If it is not technically possible or if it does not appear scientifically necessary to give information on one or more of the items below, the reasons shall be clearly stated.

2.1. Name or other identifier of each substance

- *2.1.1. Name(s) in the IUPAC nomenclature or other international chemical name(s)*
- *2.1.2. Other names (usual name, trade name, abbreviation)*
- *2.1.3. EINECS or ELINCs number (if available and appropriate)*
- *2.1.4. CAS name and CAS number (if available)*
- *2.1.5. Other identity code (if available)*

2.2. Information related to molecular and structural formula of each substance

- *2.2.1. Molecular and structural formula (including Smiles notation, if available)*
- *2.2.2. Information on optical activity and typical ratio of (stereo) isomers (if applicable and appropriate)*
- *2.2.3. Molecular weight or molecular weight range*

2.3. Composition of each substance

- *2.3.1. Degree of purity (%)*
- *2.3.2. Nature of impurities, including isomers and by-products*
- *2.3.3. Percentage of (significant) main impurities*
- *2.3.4. Nature and order of magnitude (… ppm, … %) of any additives (e.g. stabilising agents or inhibitors)*
- *2.3.5. Spectral data (ultra-violet, infra-red, nuclear magnetic resonance or mass spectrum)*
- *2.3.6. High-pressure liquid chromatogram, gas chromatogram*
- *2.3.7. Description of the analytical methods or the appropriate bibliographical references for the identification of the substance and, where appropriate, for the identification of impurities and additives. This information shall be sufficient to allow the methods to be reproduced.*"