IUCLID 5 COMPOSITION AND ANALYSIS GUIDANCE DOCUMENT:

IRON ORES, AGGLOMERATES [EINECS NUMBER 265-996-3, CAS NUMBER 65996-65-8] - IRON ORE PELLETS

INTRODUCTION

Each REACH registrant is required to file its own IUCLID 5 dossier. The IUCLID 5 dossier consists of 14 sections, of which sections 1 and 3 have to be completed by individual registrants [the other sections will be completed by the Lead Registrant and will be linked to the dossiers of all registrants who join the Joint Submission]. *Our guidance document on completion of IUCLID 5 sections 1 and 3 for iron ore pellets will be available soon and will complement this document.*

This document deals with section 1.4 of the IUCLID 5 dossier - see Figures 1 and 2. This information has to be provided by all member registrants.

Figure 1: Screenshot of IUCLID 5 section 1.4 - top

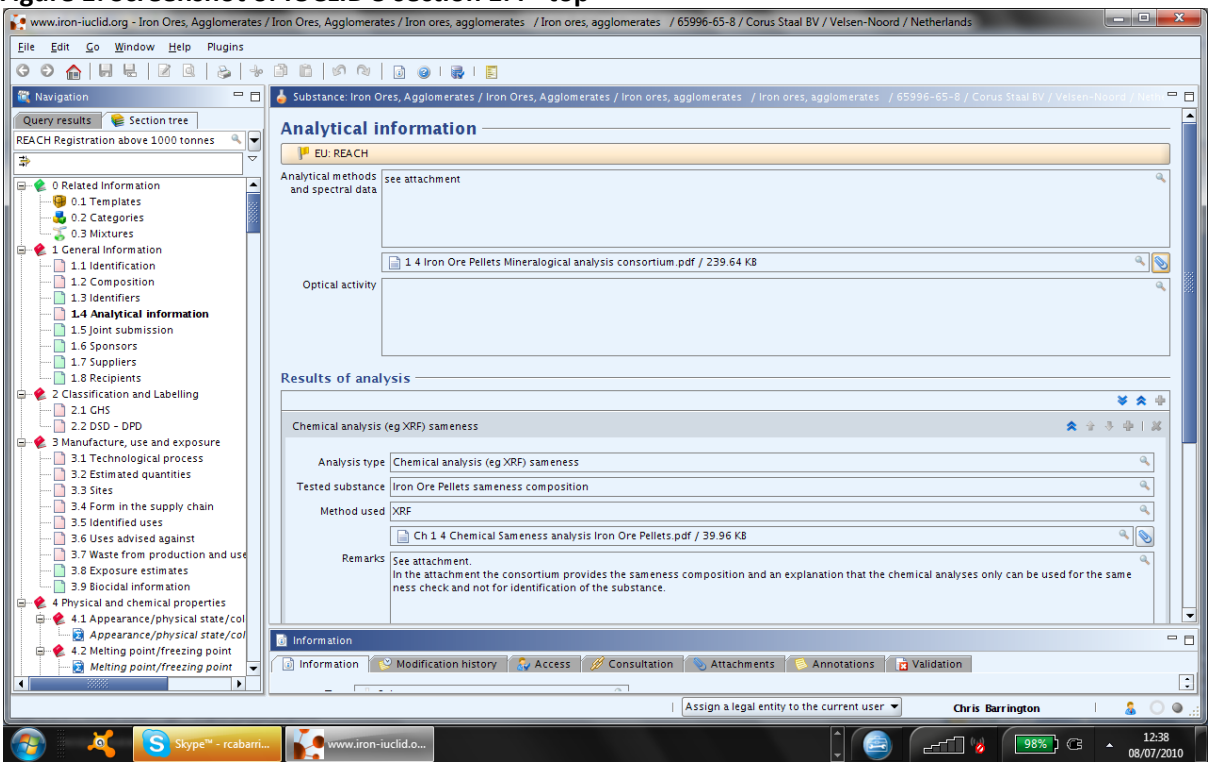
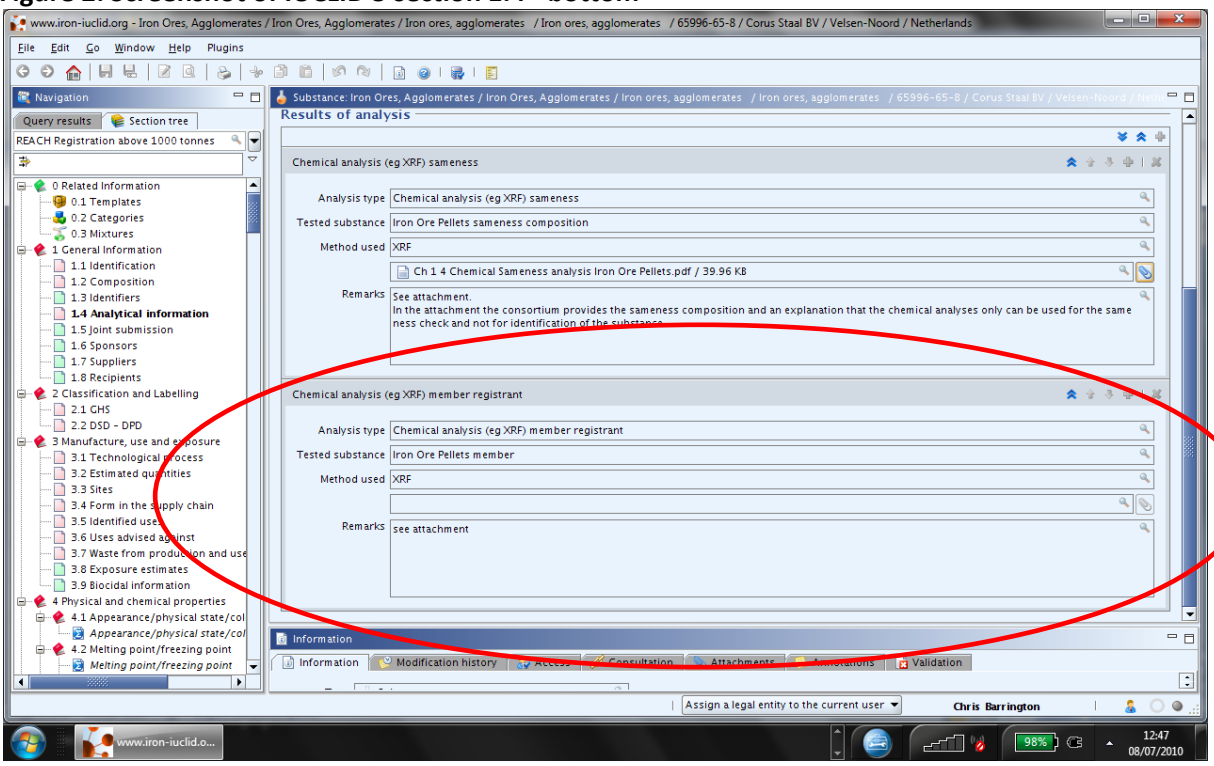


Figure 2: Screenshot of IUCLID 5 section 1.4 - bottom



It is the responsibility of member registrants to ensure that their substances are in compliance with the sameness specification entered by the LEAD Registrant in its dossier [see Table 1 below]. In simple terms, ECHA needs evidence to be able to assess that the substance in each member registrant's registration dossier is the same as that submitted by the Lead Registrant in the Joint Submission.

Annex VI, Section 2.3 of the REACH legislation specifies certain analytical evidence to be submitted in each individual registration that will generate a "fingerprint" of the substance concerned. This fingerprint will demonstrate that the substance registered is the same as that in the Joint Submission.

Each registrant is responsible for deciding how they are going to satisfy this Analytical Data requirement - this document provides the recommendations of the Iron Platform in this respect.

The Lead Registrant will include the following information in section 1.4 of its IUCLID % dossier:

- Chemical sameness criteria based upon the bulk chemical composition of iron ore pellets;
- Particle size sameness criteria for iron ore pellets;
- X-ray diffraction data to demonstrate the typical mineralogical phases present in iron ore pellets.

The Lead Registrant will make its XRD data available to member registrants for the purposes of proving sameness.

SAMENESS

The term iron ore pellets refers to the thermally agglomerated substance formed by heating a variable mixture of iron ores, limestone, olivine, bentonite, dolomite and miscellaneous iron-bearing materials in the temperature range 1250 to 1350°C. The iron ore pellets are used principally as a burden material in the production of iron in the blast furnace. The identity of iron ore pellets is summarised in Table 1 below.

Table 1: Identity of Iron Ore Pellets

Chemical name	Iron Ores, Agglomerates
IUPAC name	
Other names (usual name, trade name, abbreviation)	Iron ore pellets, iron oxide pellets
EINECS No.	265-996-3
CAS name and CAS No.	65996-65-8
Other identity code: Related CAS No.	Hematite (Fe_2O_3) 1317-60-8
Molecular formula	Fe_2O_3
Structural information (Crystal lattice)	
Minerals of identical or similar composition	Hematite
MW (g/mole)	159.7

The typical specification for Iron Ore Pellets is given in Table 2 below. Many pellet manufacturers screen their pellets to remove fines prior to loading on board vessels or trains for delivery to customers. The resultant screened off fines are known as pellet fines [also as pellet screenings or pellet chips]. The sameness specification for pellet fines is the same as that for pellets, except for nominal size, and pellet fines can be considered as pellets for the purposes of REACH.

Table 2: Sameness Specification for Iron Ore Pellets

Constituent	**Typical range, % m/m**
Fe_2O_3	>80
Fe	60-69
SiO_2	<10
Al_2O_3	<3
CaO	<8
MgO	<5
P	<0.2
S	<0.1
Free moisture at 105°C	<5
Nominal size - iron ore pellets	5-20 mm (undersize <5%)
Nominal size - pellet fines	<10 mm

It is conventional to represent the bulk composition of complex oxide materials, such as iron ore pellets, iron sinter, minerals, ores and refractory products, in terms of the simple oxides of the constituent elements, as shown in the chemical analysis in Table 2. However, this does not imply that the product is composed of a mixture of such simple compounds. It is simply a convenient

means of representing the overall elemental composition of the material with each element concentration expressed in the form of its stable oxide.

ANALYSIS TECHNIQUES

The bulk chemical compositional analysis of iron ore pellets is normally carried out using X-ray fluorescence [XRF] spectrometry by the fused bead technique or with the original substance. For the fused bead method, typically, a 0.5 to 1-g portion of finely ground and ignited iron ore pellets is mixed with alkali borate [e.g. lithium metaborate] in the ratio 1:10 (sample:borate) and the mixture is fused and cast into a circular glass bead. When the original substance is used, it is finely ground and mixed with a binding agent. In either instance the resultant test sample is subsequently subjected to multi-element analysis by XRF spectrometry using well established calibration.

There are no specific EN standards for the multi-element analysis of iron ore pellets by XRF spectrometry. However, ISO standard 9516-1:2003 for the analysis of iron ore [Iron ores – determination of various elements by X-ray fluorescence spectrometry – Part 1: Comprehensive procedure] is applicable to iron ore pellets. Laboratories undertaking such analyses should hold accreditation to ISO 17025 or ISO 9001.

Multi-element analysis of iron ore pellets provides the overall concentrations of the main constituents of the product, but does not give any indication of the identity of the individual compounds or chemical phases present, i.e. the pellet mineralogy. Quantitative phase analysis of all the major chemical phases present in iron ore pellets can be achieved only by means of X-ray diffraction [XRD] analysis combined with Rietveld data analysis.

Mineralogically iron ore pellets comprise, essentially, relict (original surviving) particles of iron ore, crystalline silica and forsterite [Mg_2SiO_4], bound together by oxide bridging formed during the process. The identified mineralogical phases present in iron ore pellets are summarised in Table 3.

Table 3: Mineralogical phases present in iron ore pellets

Phase name	Assumed chemistry for XRD refinement
Hematite	Fe_2O_3
Magnetite	Fe_3O_4
Quartz	SiO_2
Cristobalite	SiO_2
Tridymite	SiO_2
Forsterite	Mg_2SiO_4

The principal variation in pellet mineralogy is in the proportion of gangue phases, such as crystalline silica and magnesium silicate [Forsterite] present in the product. These will vary depending upon the pellet feed material and the type and amount of any additives to the feed, such as limestone, dolomite, olivine, bentonite etc.

Other techniques

As mentioned above, the principal methods of analysis used for characterisation of iron ore pellets are XRF spectrometry, for bulk analysis, and XRD for phase analysis. Since iron ore pellets are inorganic materials, analytical techniques such as nuclear magnetic resonance [NMR] spectroscopy,

infra-red [IR] spectrometry and ultra-violet [UV] absorption spectro-photometry are not suitable, since these techniques are used to investigate the molecular bonding states of organic compounds that contain essentially covalent bonds. They are not appropriate methods for the identification of inorganic structures where the bonds are principally ionic or metallic in character.

Gas chromatography [GC] is also an inappropriate analytical technique for inorganic solids since it can only be applied to organic (covalent) substances that are vaporised at temperatures below ~320°C. Similarly, high-performance liquid chromatography [HPLC], which is applicable principally to organic compounds, is not a suitable method for identification of inorganic substances. Mass spectrometry can only be applied if high energy excitation techniques, such as spark discharge or laser ablation, are used to vaporise the sample for introduction into the mass spectrometer. However, these techniques essentially provide the same information as X-ray fluorescence spectrometry, which is highly developed and widely applied for product and process control purposes. Thermo-chemical methods of analysis, such differential thermal analysis [DTA], differential scanning calorimetry [DSC] or thermo-gravimetric analysis [TGA) may be applied for specific investigations on chemical phase changes and chemical reactions that occur when iron or iron oxide materials are heated, but the data provided by these techniques are not generally sufficient for identification of these materials.

ICP-AES [inductively coupled plasma - atomic emission spectrometry], ICP-MS [inductively coupled plasma-mass spectrometry] or AAS [atomic absorption spectrometry] methods may be used for the analysis of iron ore pellets, but these techniques are generally more time-consuming and laborious than XRF spectrometry. Moessbauer spectroscopy is a useful technique for the identification and quantification of iron-bearing phases [Fe_3O_4 and Fe_2O_3, etc.] in iron ore pellets, however, the technique is not commonly applied in industry since the instrumentation required is specialised and generally only available in research institutions. The technique is less useful than XRD since it does not provide information on non-ferrous phases such as silica and silicates.

As far as particle size distribution is concerned, this should be verified by means of a sieve test in compliance with an internationally accepted standard [ISO, ASTM, etc.].

In summary:

- **member registrants will need to enter their own chemical analysis data into their dossiers in order to demonstrate that they meet the analytical sameness criteria, such analysis is recommended to be carried out in accordance with this document, XRF spectrometry being the preferred method;**
- **Particle size distribution analysis should be carried out using a method that complies with the relevant standard [ISO, ASTM, etc.];**
- **The Lead Registrant will provide its XRD data to member registrants for reference purposes, but member registrants should carry out their own XRD analysis and enter it into their dossiers.**