Sample Preparation With Induction Fusion Furnaces

We do not want to regard the chemical side, but to compare the technical possibilities of the thermal fusion methods of the past with today's technics. Thereby, it is no matter whether we regard sample preparation for a glass tablet, a powdered or again pressed powder tablet, or only powder as a product of a powdered melting fusion. The subsequent utilization of induction fusion in X-ray fluorescence or, e.g. over a solution for ICP or AAS, also should not matter here.
Exact weighing technics as well as the important mixing, exact working as well as the purity of the sample and the fusion means are also condition as e.g. the drying of the samples respectively the fusion means at 105-110°C to remove the humidity of the surface. Certainly of help, where it is necessary respectively allowed, is the glowing of the sample to remove e.g. organic parts.
Fusion means dilution of a sample in elements with a low ionization potential e.g. mainly used tetraborates or carbonates of Sodium or Lithium, samples with metallic components, the hygroscopic potassium pyrosulphate. The experience shows a dilution rate of 1:3 to 1:20 to reduce interelement effects, to avoid, respectively to reduce, self absorption and especially to produce linear calibration curves. It should not specially be referred to the problem of adding buffer materials such as SrO to reduce the fusion time respectively to reduce wetting of fusion crucibles.

The main requirements for an induction fusion furnace are :

1. accuracy 2. reproducibility 3. flexibility 4. time saving 5. operating safety 6. operating costs

If you now compare the three main fusion methods to these requirements, you will see the advantages of one method, which has of course also disadvantages, but offer such a lot of advantages that the technical and commercial decision are programmed.

I The gas-flame or the Bunsen burner

Advantages are quick readiness for working and low costs.

Disadvantages are the well-known risks at operation with open gas flames, the heat loss, the exhaust emission in the laboratory, danger of explosion.
Also, the problems of the use of crucibles made of other materials than Pt/Au as well as the crucible wear respectively the endurance have to be mentioned.
Also, automatic units show no advantages, in the opposite, there occur much more disturbances, especially on the chemical side. The temperature, even relatively, is hard to reproduce. The adjustment of the burner with gas and air as well as the situation of the crucible, the hold-up etc. are of great matter. A very great mechanical movement is necessary to achieve a mixing.

II Muffle Furnace, electrically heated

At the moment, the muffle furnace is the most frequently used fusion unit. Because of the easy controllability of the temperature as well as the universal use, the disadvantages for normal, low price muffle furnaces are mainly the low temperature for executing the fusions fast and reasonably. An essential disadvantage is the slow heating-up time and therefore the inertia until readiness for working as well as the high energy consumption at continuous operation. Of course modern, fibre-insulated laboratory furnaces with Kanthal - Super - heating offer advantages in these points, however, one has also to regard the problems of fibre materials with fusion means - steams.
The cost for such a furnace of modern technics, with electronic temperature control, stainless steel housing, fibre insulation, Kanthal - Super heating as well as exhaust are equal to those of an induction fusion unit, which is far more energy saving and operates more flexible.

III Induction Fusion

a. Principle

Induction heating units, even in the shape of a motor AC generator, a semi-conductor oscillator or the classic tube generator, work mainly according to the principle of transformers in respect to the energy transmission. The primary coil of this transformer is the induction coil of the high frequency generator of relevant frequency, the secondary turn represents the load, e.g. in this case the crucible itself. The main factor determining such a unit, is the frequency as well as the power of the high frequency generator. Due to the physical condition of the attenuation distance / skin - effect law, an operating frequency has to be chosen, which produces the necessary field for the dimensions of the crucible and if possible - in regard to the attenuation distance - is calculated this way, that the field also reaches the interior of the crucible. This is reasonable to produce ponderomotoric forces in the fusion material through electromagnetic field. Simply said, a bath movement should be achieved, mixing the fusion material in the sample at optimum. This is possible due to the conductivity of the molten glasses which is nearly in the range of bad - conducting metals. When regarding the crucible material as criterion, it relates very strongly to the frequency when using different materials e.g. from pure platinum to graphite or glass carbon. In case above occurs, in principle, it is difficult to determine the optimum work frequency. The experience and works made in praxis with different crucibles and fusion materials have shown frequencies between 500 kHz to 1,5 Mhz. In order to achieve a reasonable high fusion temperature, the high frequency generator should supply so much power at the induction coil that the radiation losses even of an uncovered, respectively uninsulated, crucible are fully covered. The field of the induction coil should be distributed so that the crucible is heated uniformly. Generally, the heating-up time to the final temperature should be 2 - 3 minutes.

b. Technics

Basically, such an induction fusion unit consists of a 1,5 kW to 2,5 kW HF-generator in tube construction, because by its high ohmic output impedance, it allows accomodation of various crucibles and induction coils without problems. A water cooled induction coil takes a ceramic or fibre material crucible. This insulation crucible serves as a radiation protection to reduce heat losses as well as an electrical insulator. The Pt/Au or graphite crucible inside of the insulation crucible is now the secondary winding in which the electro - magnetical field is turned into heat.
The HF-output can be adjusted at the primary side of the high - voltage transformer. Over the anode DC current respectively the percentual power - adjustment, also the relative temperature can be adjusted reproduceably.
Exact temperature measurements are only possible with a very high expenditure, because emission factors, production of steam as well as reflection produce great insecurity here.

If no temperature measurement is made, it is absolutely necessary to adjust the fusion time exactly. For that, a corresponding timer has to be provided.
According to the viscosity of the fusion means with the sample which is, of course, very dependant on the temperature, it can be necessary to equip the crucible with a vibration device to avoid bubbles.
An exhaust device in the fusion unit is necessary, because at the high fusion temperature steam pressures cause a corresponding impurity of the sample chamber.

c. Application

In case a platinum - gold crucible is used, the application temperature is limited to 1500°C due to the softening of the crucible material. With the presaid powers and insulation crucibles, the sample fusion mixture can be fused in approx. 3 minutes. It is recommended to heat - up slowly during the first minute to avoid bubbling of the fusion material. The fusion and melting process can be observed through a viewing glass in the cover of the unit. The fusion and even solidification can be done in the crucible, depending on the kind of crucible to be used. However, this has the disadvantage that very big crucibles have to be used which are very expensive when using platinum / gold crucibles. It seems to be much more reasonable to use the standard crucibles and casting shells. These can be preheated, either on the fusion crucible or on a separate induction coil, which can also be switched in series to the melting coil. For the last possibility, the same physical conditions are given as for the fusion crucible itself. Especially for long - lasting fusion e.g. chrome ores, high fusion temperatures and also high mould temperatures are recommended, which can only be obtained through induction heating. Losses are, compared to other fusion methods, very low, which proves the standard deviation as the fusion times are relatively short.
Swivelling of the crucible normally is not necessary, as the bath movement through the electromagnetic field as well as through the thermal flow at high temperatures is sufficient. After having finished the fusion process, the holder together with the crucible is taken out of the coil and the fusion is casted into the preheated mould. The casting process is done with no problems compared to other procedures as the viscosity is much more favourable due to the higher temperatures. The mould can be cooled through normal heat losses of through punctual cooling with compressed air from the bottom of the sample.
In any case, this way the glass buttons are nearly completely free from reams and seeds. It remains to pay attention to the construction of the casting mould where the surface - when using the buttons directly - is very strongly influenced through fractures and roughness. It still remains to say that Pt/Au or Pt/Au/Zr - crucibles without any problem 150 to 250 fusions when treating them carefully. Graphite or glass carbon withstands even when admitting argon, max. 25 to 35 fusions.

Summary

The advantages of the inductive melting fusion are the simple servicing and operation safety with higher temperature and very reduced time. A minimum total expenditure is obvious. Accuracy of analysis and economy with optimum sample quantity certify the high quality of this method, inspite of element - specific material loss, which can be rectified. A great number of technical as well as chemical use - publications have proved that.

Chromium - Magnesite - Fusion

Sample	fusion		insoluble		fused
1 g weight	technics		residue		substance(%)
1	Lifumat		0,078		92
2	(5 min.)		0,074		
3	muffle fce		0,195		77
4	(20 min.)		0,263		

Gauge slag mixing

RFA (powder briquette)		Fe	P_2O_5	SiO_2	CaO	MgO	MnO	sum	fusion technics
X_S	(%)	17,07	10,28	12,07	44,10	2,98	3,66	90,160	muffle furnace
S	(%)	0,01	0,02	0,04	0,14	0,07	0,01		
X_S	(%)	16,97	10,22	11,83	43,81	2,99	3,64	89,46	Lifumat
S	(%)	0,01	0,04	0,04	0,19	0,04	0,01		(3 min.)