

SPECTROFlux®

ALKALI BORATE FUSION FLUXES

Products
Applications
Techniques



Introduction to Alfa Aesar

Spectroflux® Fluxes

Alfa Aesar has a world-renowned reputation for consistently supplying the highest quality analytical fluxes to the cement and steel industries and geo-scientists for mineralogical applications.

For more than 30 years chemists have relied on Spectroflux alkali borate fusion fluxes for use in methods such as direct reading optical or X-ray emission spectrometers, atomic absorption spectrometers, spectrophotometers, polarographs, ion selective electrodes, inductively coupled plasma or classical analytical techniques. Our batch specific analysis ensures reproducibility and accuracy of your results.

Alfa Aesar offers a wide range of fluxes and supplies customised formulations to meet specific analytical requirements.

KEY FEATURES OF SPECTROFLUX PRODUCTS

- Consistent bulk density.
- Large batch sizes.
- Good homogeneity.
- Highest degree of purity, min 99.95%.
- Low loss on fusion.
- Available in quantities from 50g samples to 100Kg batches.
- Each batch is analysed for 27 inorganic impurities and loss on fusion.
- All orders are supplied with a batch specific certificate of analysis.
- Delivered in 1Kg or 5Kg sealed containers.
- Standard fluxes are available from stock.
- Our flux is priced competitively.

Typical Materials Analysed

Materials Analysed

The analysis of refractory materials can be reduced to a simple, accurate procedure with the use of Spectroflux analytical fluxes.

Fusion with a molten alkali metal borate flux provides a rapid and simple means of dissolving chemically stable materials to yield glass-like, solid solutions. The method eliminates any inhomogeneity of particle size, density or composition in the analytical sample taken. (See pages 6-7 for detailed sample preparation details).

Samples of a wide variety of materials may be prepared in a simple manner for analysis by instrumental or chemical techniques. Typical materials analysed using flux fusion are shown below.

- Aluminosilicate refractories
- Aluminum ores: aluminas
- Carbides
- Cement, raw mix and finished; concrete
- Chrome ores and refractories
- Coal ashes and furnace deposits
- Copper ores; slags and concentrates
- Iron ores: iron and related slags.
- Iron sinter; steel slags ferro-alloys
- Lead ores and slags
- Manganese ores and slags
- Metal alloys
- Minerals and ores
- Niobium and tantalum ores
- Rare earth ores
- Silicates and aluminosilicates
- Phosphate and carbonate rocks
- Soils
- Tin ores and concentrates
- Titanium ores
- Tungsten ores
- Welding fluxes
- Zircons: silicon and boron carbides



Spectroflux Selection

Spectroflux Selection

THE IDEAL FLUX WILL:

- Act as a solvent for a wide range of composition.
- Possess a low melting point to minimise volatilisation of flux and sample, facilitate handling, minimise power costs and prolong crucible life.
- Have a low molten viscosity to aid mixing during fusion, pouring from crucible and rapid dissolution of sample.
- Produce a transparent bead at minimum dilution with a wide range of samples free from devitrification.

- Be non-hygroscopic to aid weighing.
- Have controlled, high density particle size to aid homogeneous mixing, rapid melting and economic use of platinum labware.
- Exhibit low loss of fusion, i.e. have low water content and be non-volatile to avoid excessive correction and pre-firings.

OUR FLUXES ARE CATERGORISED WITHIN THREE RANGES:

- "Classic" Spectroflux
- New prefused fluxes
- Specialised and custom fluxes



Spectroflux Selection

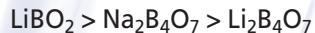
"Classic" Spectroflux Products

Spectroflux Selection

Cat#	Name	Composition	w/w%	Mpt °C	Density g/cm ³	Typical Applications
12078	100	Lithium tetraborate	100	920	>0.5	Cement, most ores, carbonates, aluminosilicates
41951	100	Lithium tetraborate (low phosphorus)	100	920	>0.5	A general purpose flux. Dissolves most refractories. Not suitable for acidic samples. Low phosphorus (20 ppm).
12079	100A	Lithium metaborate	100	845	>0.5	Sulphates, phosphates, silicas, sands, clays
12080	100B	Lithium tetraborate Lithium metaborate	20 80	830	>0.5	Aluminosilicate range, aluminas, borax frits, cements, iron, blast slags
97890	100D	Lithium tetraborate Lithium metaborate	35 65	825	>0.5	Aluminosilicate, aluminas, bauxites, iron ores
12087	110A	Lithium tetraborate Lithium metaborate	50 50	870	>0.5	Silicates, calcareous materials, chrome ores, sands & shales
12086	110	Lithium tetraborate Lithium metaborate	66.5 33.5	875	>0.5	Cement products, aluminosilicates, calcareous refractories
36222	120A	Lithium tetraborate Lithium fluoride	90 10	780	>0.5	Petroleum
12321	106	Lithium tetraborate Lanthanum oxide	85 15	900	>1.5	Steel, sinters, slags, cements, phosphates, carbonates
12320	107	Lithium tetraborate Lanthanum oxide	81.8 18.2	900	>1.5	Cement

- The ratios of the homogeneous blends can be adapted to your specific needs
- A low phosphorus version of SF100, below 20 ppm is available on request

FLUX SELECTION is dependent on the acidity/basicity of both sample and Spectroflux analytical flux. In decreasing order of basicity, the pure alkali metal borate fluxes can be grouped as follows.



Samples can be broadly grouped into three categories:

Acidic

SiO_2 , P_2O_5

Basic

M_2O , MO or M_2O_3 (M = Metal such as Na_2O , MgO or Al_2O_3)

Amphoteric

Fe_2O_3

Most flux fusion is carried out with lithium tetraborate, lithium metaborate or a blend of each. The addition of lithium metaborate reduces the fusion point of the melt.



Spectroflux Selection

Application Notes on "Classic" Spectroflux Products

SF100 (Li₂B₄O₇)

Lithium tetraborate is an 'acidic' flux suited to dissolution of samples containing a high concentration of non-metallic basic oxides, carbonate rocks and aluminosilicates. High silica bearing samples dissolve slowly (1h); in contrast with aluminas and aluminosilicates (20 min) at 1,200°C. Beads produced from Li₂B₄O₇, which are significantly less hygroscopic than those prepared using Na₂B₄O₇, do not generate interfering X-ray emissions, and have a lower absorption for light element radiations. By using Li₂B₄O₇, sodium can be included in the analytical program.

SF100A (LiBO₂)

Lithium metaborate is a basic flux. It rapidly dissolves a wide range of aluminosilicates, in particular, the more acidic oxide compositions. However, materials containing in excess of 85% Al₂O₃ tend to promote devitrification of the bead, due to undissolved particulates. LiBO₂ is generally used when an aqueous medium is required for analysis, e.g. AA spectrometry.

Due to its relatively low melting point, fusion can be effected over a gas burner and the melt is free-flowing even at 1,000°C. LiBO₂ will not cast in the absence of a sample to give a clear transparent bead. Beads are less likely to crack than those prepared using Li₂B₄O₇.

SF100B (Li₂B₄O₇:4LiBO₂)

A eutectic composition specially formulated to dissolve aluminosilicates ranging from 100% SiO₂ to 100% Al₂O₃. It combines the superior melt characteristics of LiBO₂ with the ability of Li₂B₄O₇ to dissolve high alumina samples rapidly. Strongly basic materials such as magnesite and calcite, when fused with 100B, will not produce stable beads.

SF100D (Li₂B₄O₇:2LiBO₂)

Formulated specifically for dissolving iron ores.

SF110 & 110A (Li₂B₄O₇:LiBO₂ homogeneous blends)

Formulated for silicate and calcareous materials, these compositions are more acidic than Spectroflux 100B and 100D

SF120A (9Li₂B₄O₇:LiF)

The Lithium tetraborate has a reduced melting point due to the presence of alkali metal fluoride. Fusions using LiF should be carried out in a well-ventilated fume cupboard.

SF106 & SF107

Lithium tetraborate with Lanthanum oxide addition. Lanthanum oxide acts as a heavy absorber maximising the sensitivity of the spectrometer.



Spectroflux Selection

New “Prefused” Spectroflux Products

Spectroflux Selection

KEY FEATURES OF NEW PREFUSED SPECTROFLUX PRODUCTS

- Free flowing, which facilitates use with automatic dispensing devices.
- Prefused dust free granules.
- Anhydrous, giving low loss on fusion.
- High bulk density, typically 1.0g/cm³ or above.
- Good homogeneity.
- Controlled particle size.
- High degree of purity.

Cat#	Name	Composition	w/w%	Mpt °C	Density g/cm ³	Typical Applications
96569	1000	Lithium tetraborate	100	920	>1.0	Carbonates, aluminosilicates
96706	1000A	Lithium metaborate	100	845	>1.0	Sulphates, phosphates, silicas, sands, clays
96689	1000B	Lithium tetraborate Lithium metaborate	20 80	840	>1.0	Aluminosilicate range, aluminas, borax frits, cements, iron, blast slags
96713	1000D	Lithium tetraborate Lithium metaborate	35 65	825	>1.0	Aluminosilicate, aluminas, bauxites, iron ores
96571	1010	Lithium tetraborate Lithium metaborate	66.5 33.5	875	>1.0	Cement products, aluminosilicates, calcareous refractories
96572	1010A	Lithium tetraborate Lithium metaborate	50 50	870	>1.0	Silicates, calcareous materials, chrome ores, sands & shales
96712	1020A	Lithium tetraborate Lithium fluoride	90 10	780	>1.0	Petroleum

Developments in our manufacturing capability have led to the introduction of our new Spectroflux prefused flux range.

It has a higher tap density (>1.0 g/cm³) than our traditional flux, reducing the volume of flux in the platinum crucible.

A small amount (0.5%-0.2%) of a non wetting agent, KI or LiI can be added to allow for easy removal of the glass disk from the platinum mould.



Spectroflux Selection

Specialised Spectroflux Products

Spectroflux Selection

Cat#	Name	Composition	w/w%	Mpt °C	Density g/cm ³	Typical Applications
12082	104	Lithium tetraborate Lithium carbonate	55.5 45.5	740	>0.7	Basic oxidising flux for sulphates, phosphates, metals, lead & titanium ores
12085	108	Lithium tetraborate Lanthanum oxide Sodium nitrate	76.2 14.3 9.5	790	>0.7	Strongly oxidising acidic flux for coal ashes & furnace deposits
96153	112	Lithium tetraborate Lanthanum oxide Lithium iodide	82 15 31	875	>1.3	Specialised applications
12088	118	Lithium tetraborate Sodium nitrate	75 25	680	>0.7	Strongly oxidising acidic flux for ferroalloys, metals, coal ashes & sulphides
12092	161	Lithium tetraborate Lithium nitrate	90 10	870	>0.7	Basic oxidising flux for ferroalloys, iron ores & slags
96714	310	Lithium tetraborate Lithium metaborate Lithium metaphosphate	17.5 17.5 65	630	>1.0	Specialised applications, petroleum
97429	125	Lithium tetraborate Lithium metaborate Lanthanum oxide Lithium fluoride	51 27 12 10	725	>0.7	Specialised applications
12093	200	Sodium tetraborate	100	740	>1.2	Iron ores, chrome refractories, rare earth minerals, tin & titanium ores

WE CUSTOM MAKE FLUXES TO MEET CUSTOMERS NEEDS, WE HAVE OVER 100 UNLISTED SPECIAL FLUXES.

SF104

Lithium carbonate dissolves acidic oxides easier than lithium tetraborate on its own. It also produces bubbles in the melt for a better mix. It also initiates the decomposition of nitrates.

SF 108, 118, 161

Based on an alkali metal borate, each of these fluxes contains lanthanum oxide as a heavy absorber to minimise inter-element effects, thus allowing the analysis of a wide range of materials on the same calibration curves. Sensitivity is slightly reduced for light elements; overlap of NaK line with LaM line occurs and minor interferences are experienced in determining Mg, Ti and Mn. Lanthanum Oxide (La_2O_3) increases the basicity of the flux and assists in the formation of a glass.

SF108, 118, 161

Based on an alkali metal borate, each of these fluxes contains nitrate or carbonate additions to provide an oxidising flux for use with samples containing reduced species. Oxidisers such as nitrates of lithium and sodium can minimise the corrosion of platinum crucibles.

SF 200

$Na_2B_4O_7$ less acidic than lithium tetraborate.



Analytical Techniques

Analytical Techniques

Spectroflux analytical fluxes can be used in conjunction with direct reading optical or X-ray fluorescence (XRF) spectrometers, atomic absorption (AA) spectrometers, inductively coupled plasma (ICP) atomic emission spectrometry, spectrophotometers, polarographs, ion selective electrodes or classical analytical techniques. Whichever method is chosen, Spectroflux analytical fluxes offer the analyst the benefits of speed and analytical precision.

SAMPLE PREPARATION GUIDELINES

Many different procedures have been described in the literature for preparation of solid solution bead samples, suitable for XRF analysis.

The general method for the preparation of materials for XRF analysis involves taking known weights of the flux and sample in an appropriate ratio (e.g. 10:1), then fusing and allowing the melt to cool to produce a stable, transparent, homogeneous and crack-free bead.

- To ensure homogeneous mixing the samples should be ground to a particle size of $<100\mu\text{m}$. To obtain the same ratio of flux to sample for each separate analysis, it is essential to ignite the sample before weighing. The weight of the flux used should then be adjusted to compensate for the observed loss on ignition of the sample. Alternatively a mathematical correction may be applied.
- The flux should also be ignited at about 700°C to remove any moisture absorbed during storage.
- The ignited sample and flux are transferred into a non-wetting platinum alloy crucible and the contents are thoroughly mixed with a chemically inert rod. The uncovered crucible is placed in a muffle furnace or over a gas burner at 900°C to $1,150^\circ\text{C}$ and swirled occasionally until the mixture is completely molten and homogeneous. Samples containing high concentrations of alumina and zirconia necessitate heating at $1,200^\circ\text{C}$.

- A platinum alloy casting dish mounted on a ceramic support is heated at the same temperature as the crucible for 2 minutes (5 to 10 minutes for aluminas and zirconias). After removal from the furnace the molten mixture is poured from the crucible into the casting dish. Once the bead has solidified, a jet of air is directed at the base of the casting dish to cool the bead.
- Spectrofluxes have been used successfully with commercially available automatic fusion equipment such as the Philips MagiX, Perl'X, PW 1400, LECO, Claisse fluxy, M4, Diano 8000 and others.



Techniques for Specific Materials

Specific Materials

Where samples are particularly difficult to dissolve, oxidising agents such as lithium or sodium nitrate may be added to the flux to speed up the dissolution of the sample.

Samples that contain sulphides should be roasted in a ceramic crucible in air, before being mixed with the flux, or fused with the flux in the presence of sodium or lithium nitrate. This ensures conversion of sulphides to sulphates and their retention in the solid solution. Sulphur is lost from fusion in graphite crucibles and when using ammonium nitrate as an oxidising agent.

Samples containing large amounts of organic matter or carbon should be ignited in air at 500°C for several hours prior to fusion.

Ferro-alloy samples must be fully oxidised prior to fusion with flux. Using lithium tetraborate, in-situ with an oxidising mixture, avoids pre-oxidation of samples, such as steel plant dust and refractories containing metals.

Samples that need to be fused at 1,200°C, e.g. aluminas, must be fused for the same period of time due to the loss of flux that occurs by volatilisation. It is also possible to compensate for losses on fusion by adding an internal standard to the flux/sample mixture.

Using a flux containing a small quantity of the halo-acids, HBr and HI, or alkali metal can enhance the non-wetting properties of 5% Au-Pt crucibles

Chrome-bearing materials containing up to 50% Cr₂O₃ may be dissolved in a mixture comprising 5Li₂B₄O₇:5LiBO₂:0.4 sample.



Analytical Instruments that use Spectroflux

Analytical Instruments

X-RAY FLUORESCENCE SPECTROMETRY (XRF)

Precision is a significant feature of X-ray spectrometry. However, the spectrometer can only yield accurate analyses if systematic errors associated with the sample are eliminated. Errors due to mineralogical, particle size and surface finish effects must be minimised. Refractory materials are particularly heterogeneous and fusion with Spectroflux analytical fluxes provides the simplest method of eliminating mineral identity and particle size interference, while reducing inter-element effects.



The methods employed to compensate for inter-element effects are:

- i) The use of mathematical corrections to compensate for enhancement and absorption
- ii) Calibration over narrow concentration ranges using closely matched standards
- iii) The use of multiple dilution with a flux
- iv) Incorporation of a strong absorber, e.g. Lanthanum oxide (La_2O_3), into the solid solution at concentrations such that variations in sample composition have little effect on the total absorption of the matrix for the elements under analysis

Flux fusion techniques therefore have a major role to play in eliminating the various interference effects.

ATOMIC ABSORPTION SPECTROPHOTOMETRY (AA)

In the analysis of silicate rocks and minerals by AA spectrophotometry, borate flux fusion is an excellent method of sample decomposition, since it is rapid and applicable over a wide range of sample compositions. The method requires no chemical separations and enables a large number of elements to be determined in a single-fluxed sample.

INDUCTIVELY COUPLED PLASMA SPECTROPHOTOMETRY (ICP)

ICP is widely used in routine trace analyses in cements and refractories. Borate fluxes are used in the normal way to provide a melt, which can be either directly dissolved into solution, or cooled and then dissolved in the relevant acid.



Platinum Labware

Platinum Labware

95% platinum 5% gold is commonly used in sample preparations of glass beads for X-ray fluorescence analysis in the glass, cement and ceramic industries. This is because of the non-wetting property, conferred on the platinum by the gold addition, which results in easy removal of samples after fusion.

Alfa Aesar offers extra strength and durability in its exclusive ZGS 95% platinum 5% gold. The ZGS is much stronger and is resistant to low level contamination. It maintains a more consistent surface, ideal for casting beads.

All types of crucibles with lids, moulds, tongs and casting dishes /lids for automatic fusion equipment can also be supplied by Alfa Aesar.

We will quote your new platinum labware at the best daily rate.

Please send us your old platinum labware. We will recycle and credit your account.



SPECTROFlux®

**Spectroflux is available from stock throughout the world.
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