Alfa Aesar®
Johnson Matthey

SPECTROFlux®

Analytical Fluxes Analytische Flussmittel Flux Analytiques

SPECTROFLUX®

Analytical Fluxes Analytische Flussmittel Flux Analytiques

CONTENTS

Alfa Aesar®
Johnson Matthey

For years chemists have relied on **Spectroflux** alkali borate fusion fluxes in analysing a wide range of refractory materials. **Spectroflux** fluxes are known for their controlled particle size, high bulk density and homogeneity.

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

A wide range of fluxes is available 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.

Regardless of the method chosen, **Spectroflux** analytical fluxes offer the benefits of speed and analytical precision. Custom formulations are available on request.

Spectroflux is manufactured at our Royston plant and is available in quantities from 50g samples to multi-kilogram batches, thus ensuring reproducibility and the accuracy of your results.

Cat. No.	Flux No.	Composition	W/W %*	Mpt °C**	Typical Applications
12078	100	Lithium tetraborate	100	920	An acidic flux for fusing basic oxides, carbonates, alumino silicates, concrete mixes, soils, magnesites, bauxites & rare earth oxides.
21789	100/ 100-500µm	Lithium tetraborate (100-500mm)	100	920	As above.
21790	100/ <100µm	Lithium tetraborate	100	920	As above.
41951	100/ low P	Lithium tetraborate (low phosphorous)	100	920	A general purpose flux with a low phosphorous content. Dissolves most refractories. Not suitable for highly acidic samples.
12079	100A	Lithium metaborate	100	845	A basic flux for fusing acidic oxides, silicas, sands, clays & spinel glasses.
573100	100A/ coarse	Lithium metaborate	100	845	As above.
12080	100B	Lithium tetraborate Lithium metaborate	80 20	840	Eutectic composition suitable for fusing the entire aluminosilicate range; aluminas, borax frits, cements, iron & blast slags.
21791	100D	Lithium tetraborate Lithium metaborate	35.3 64.7		Specialised applications.
21792	100E	Flux 100D Sodium Nitrate***	95 5		Specialised applications.
12082	104	Lithium tetraborate Lithium carbonate***	55.5 45.5	740	A basic oxidising flux for fusion of samples with traces of reduced species. Suitable for sulfates, phosphates, metals, alloys, acidic minerals, lead & titanium ores.
12083	105	Lithium tetraborate Lithium carbonate Lanthanum oxide	47.0 36.7 16.3	700	Similar to flux 104 incorporating lanthanum oxide as a heavy absorber. Used where computer signal processing is limited.
12321	106	Lithium tetraborate Lanthanum oxide	85 15	900	A similar flux to 100 containing lanthanum oxide as a heavy absorber; sinters, slags, cements, phosphates & carbonates.
12320	107	Lithium tetraborate Lithium oxide	81.8 18.2	900	Specialised applications.
12085	108	Lithium tetraborate Lanthanum oxide Sodium nitrate***	76.2 14.3 9.5	790	A strongly oxidising acidic flux for use with samples containing significant amounts of reduced species-coal ashes & furnace deposits.
12086	110	Lithium tetraborate Lithium metaborate	66.5 33.5	875	Intermediate acidity. Suitable for aluminosilicates & calcareous refractories.
12087	110A	Lithium tetraborate Lithium metaborate	50 50	870	More acidic than flux 100B, suitable for fusion of silicates, calcareous materials, chrome ores, sands & shales.
21793	112	Lithium tetraborate Lanthanum oxide Lithium iodide	82 15 3		Specialised applications.
12088	118	Lithium tetraborate Sodium nitrate***	75 25	680	A strongly oxidising acidic flux suitable for samples containing significant amounts of reduced species; ferroalloys, metals, coal ashes & sulphides.
12089	120	Lithium tetraborate Lithium fluoride***	80 20	780	A low viscosity, low melting, acidic flux with a lower melting point than flux 100 & improved better non-wetting properties, suitable for slags, ores & refractory silicates.
36222	120A	Lithium tetraborate Lithium fluoride***	90 10	780	Specialised applications.
12090	128	Lithium tetraborate Lithium metaborate Sodium nitrate***	33.0 61.5 5.5	840	A basic oxidising flux for use with acidic samples containing reduced species. Beads water soluble for use with ICP, AA techniques.
12092	161	Lithium tetraborate Lithium nitrate***	90 10	870	A basic oxidising flux for use with samples containing a high level of reduced species; ferroalloys, iron ores & slags.
12093	200	Sodium tetraborate	100	740	The sodium analogue of flux 100 for iron, iron ores, chrome refractories, rare earth minerals, tin & titanium ores.

---

* Composition prior to firing is quoted in cases where a flux is prepared from a compound containing water of crystallisation or carbonate ion.
**Average melting point over which the material melts. The fluid temperature of most fluxes is 200 to 300°C higher than the average.
*** Compound added after fusion of other constituents.

SPECTROFLUX® ANALYTICAL FLUXES

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

Refractory materials such as ores, slags and minerals are often very resistant to acid attack making the preparation of a sample solution difficult and time consuming. Selection of an effective method of disintegrating the sample matrix to a reproducible form is a prerequisite for speedy, precise and accurate analysis. Flux fusion is such a method.

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.

Samples of a wide variety of materials may thus be prepared in a simple manner for analysis by instrumental or chemical 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 analysis involves taking known weights of the flux and sample in an appropriate ratio (e.g. 10:1), fusing and allowing the melt to cool to produce a stable, transparent, homogeneous and crack-free bead. To ensure that the same ratio of flux to sample is used 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°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°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

i) 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.

ii) 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 fusions in graphite crucibles and when using ammonium nitrate as an oxidising agent.

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

iv) 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.

v) Samples which 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.

vi) The non-wetting properties of 5% Au-Pt crucibles can be enhanced by using a flux containing a small quantity of the halo-acids, HBr and HI, or alkali metal bromide or iodide. Halides increase the fluidity of the melt, aiding both mixing and removal of molten mixtures. This is particularly useful in the case of copper, cobalt and chromium bearing samples, which are prone to sticking.

vii) Chrome-bearing materials containing up to 50% Cr_2O_3 may be dissolved in a mixture comprising $5Li_2B_4O_7$:$5LiBO_2$:0.4 sample.

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.

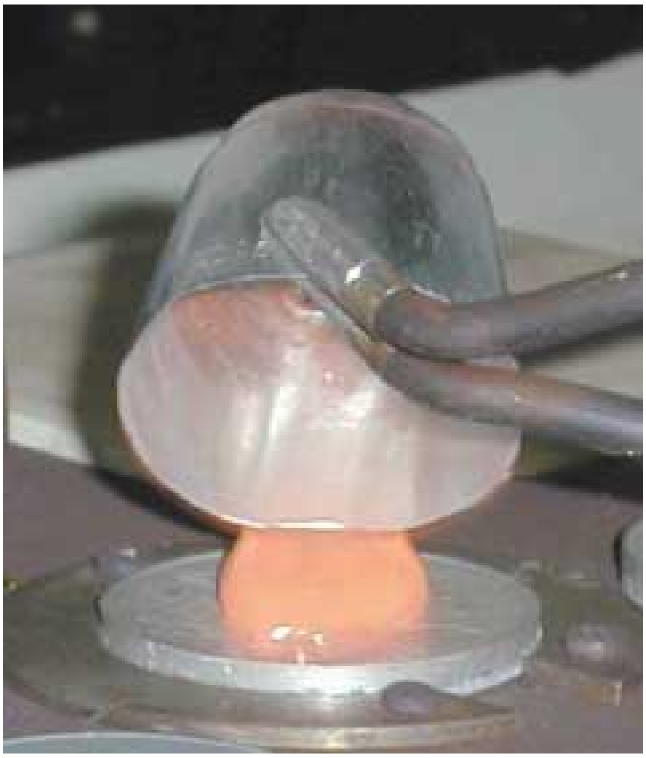
The methods employed to compensate for inter-element effects are:

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.

CHEMICAL ANALYSIS

Flux fusion and dissolution of the resulting solid solution in dilute acid provide a rapid means of obtaining a sample solution for chemical analysis of materials resistant to direct acid attack. When a sample has been fused with a borate flux and if the presence of boron interferes with the analytical procedure, the boron can be removed by repeated evaporation with methanol, saturated with hydrogen chloride. This method of sample preparation has also been successfully employed in spectrophotometric and polarographic analyses.

OPTICAL EMISSION SPECTROGRAPHY

Borate fluxes perform a dual function in optical emission spectrographic analysis. Firstly, they provide simple and effective means of disintegrating the sample into a glass-like solid of uniform composition. Referred to as ‘iso-formation’, this ensures that the same type of sample is always presented to the spectrograph. Secondly, a borate flux acts as a spectrochemical ‘buffer’, so that the spectral emission of the elements under examination are not affected by variations in sample composition. Alkali-metal borates are particularly useful as spectrochemical buffers since their spectra introduce very little line interference. Inter-element effects can be minimised by the use of a flux containing internal standards, e.g. cobalt or strontium.

FLUX SELECTION

The ideal flux will:

- act as a solvent for a range of compositions
- possess a low melting point to minimise volatilisation of flux and sample, facilitate handling, minimise power costs and prolong crucible life
- possess low viscosity when molten 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 and storage
- 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 expertise in this area has led to improvements from which you can benefit. The preparative routes used by Alfa Aesar are designed to yield Spectroflux analytical fluxes that incorporate:

- low loss on fusion
- controlled particle size
- high bulk density
- homogeneity
- low levels of impurity elements

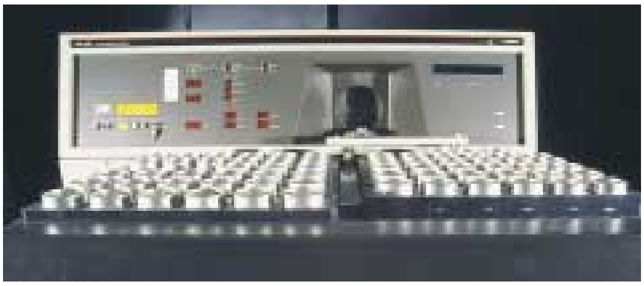
Alfa Aesar offers a complete range of fluxes and regularly honours requests for customised formulations to meet your specific analytical requirements.

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.

$LiBO_2 > Na_2B_4O_7 > Li_2B_4O_7$

Samples can be broadly grouped into three categories:

Acidic	e.g. SiO_2, P_2O_5
Basic	e.g. M_2O, MO or M_2O_3 (M = Metal) such as Na_2O, MgO or Al_2O_3
Ampohoteric	Fe_2O_3



SPECTROFLUX 100 Lithium tetraborate ($Li_2B_4O_7$)

An 'acidic' flux suited to dissolution of samples containing a high concentration of 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 $Li_2B_4O_7$, which are significantly less hygroscopic than those prepared using $Na_2B_4O_7$, do not generate interfering X-ray emissions, and have a lower absorption for light element radiations. By using $Li_2B_4O_7$, sodium can be included in the analytical program.

SPECTROFLUX 100A Lithium metaborate ($LiBO_2$)

Rapidly dissolves a wide range of aluminosilicates, in particular, the more acidic oxide compositions. However, materials containing in excess of 85% Al_2O_3 tend to promote devitrification of the bead, due to undissolved particulates. $LiBO_2$ 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. $LiBO_2$ will not cast in the absence of a sample to give a clear transparent bead. Beads made with $LiBO_2$ are less likely to crack than those prepared using $Li_2B_4O_7$.

SPECTROFLUX 100B ($4LiBO_2$:$1Li_2B_4O_7$)

An eutectic composition specially formulated to dissolve aluminosilicates ranging from 100% SiO_2 to 100% Al_2O_3. It combines the superior melt characteristics of $LiBO_2$ with the ability of $Li_2B_4O_7$ to dissolve high alumina samples rapidly. Strongly basic materials such as magnesite and calcite, when fused with 100B, will not produce stable beads.

SPECTROFLUX 105, 106, 108, & 112

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.

SPECTROFLUX 100E, 104, 108, 118, 128 & 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.

SPECTROFLUX 110 & 110A

Formulated for silicate and calcareous materials, this composition is more acidic than Spectroflux 100B.

SPECTROFLUX 120 & 120A

Both compositions have a relatively low melting point due to the presence of alkali metal fluoride. Fusions using these compositions should be carried out in a well-ventilated fume cupboard.

SPECTROFLUX 200 SERIES

These are the sodium analogues of the Spectroflux 100 series. Sodium borate beads are hygroscopic by nature.

SPECIAL FLUXES

Alfa Aesar will gladly consider producing fluxes to suit customers' needs. We already have over 100 unlisted special fluxes!

FLUX FUSION: TYPICAL APPLICATIONS

Spectroflux analytical fluxes are used throughout industry for effecting rapid dissolution of many different types of chemically stable inorganic materials. Typical materials analysed using a flux fusion decomposition technique are shown below.

- aluminosilicate refractories
- aluminum ores: aluminas
- carbides
- cement, raw mix and finished; concrete
- catalyst supports
- chrome ores and refractories
- coal ashes and furnace deposits
- copper ores; slags and concentrates
- iron ores: iron and related slags; iron sinters; steel slags ferro-alloys
- lead ores and slags
- manganese ores and slags
- metal alloys
- niobium and tantalum ores
- rare earth ores
- silicate; aluminosilicate; phosphate and carbonate rocks; minerals and ores
- soils
- tin ores and concentrates
- titanium ores
- tungsten ores
- welding fluxes
- zircons: silicon and boron carbides

PLATINUM ALLOY CRUCIBLES

Unalloyed platinum and graphite crucibles are quite often used to fuse samples of refractory materials but both suffer from the disadvantage that they are wetted by the fusion mixture.

To overcome this disadvantage, Johnson Matthey has developed a special range of platinum laboratory apparatus including gold/platinum, gold/rhodium/platinum and the highly resistant zirconia grain stabilised platinum (ZGS) alloy. All types of casting dishes and lids for automatic fusion equipment can also be supplied by Johnson Matthey.

Pictures courtesy of Department of Geology, Royal Holloway, University of London and CERAM Research Limited

Alfa Aesar
Johnson Matthey

Europe Alfa Aesar • Johnson Matthey GmbH • Postfach 11 07 65 • D-76045 Karlsruhe GERMANY
Free Phone: 00-800-4566-4566 or +49-721-84007-280 • Free Fax: 00-800-4577-4577 or +49-721-84007-300
E-Mail: special@matthey.com • www.alfa-chemcat.com

United States Alfa Aesar • A Johnson Matthey Company • 30 Bond Street • Ward Hill, MA 01835 - 8099 USA
Phone: 1-888-343-8025 or 1-978-521-6401 • Fax: 1-978-521-6366
E-Mail: specials@alfa.com • www.alfa.com