ILUKA'S SYNTHETIC RUTILE PRODUCTION

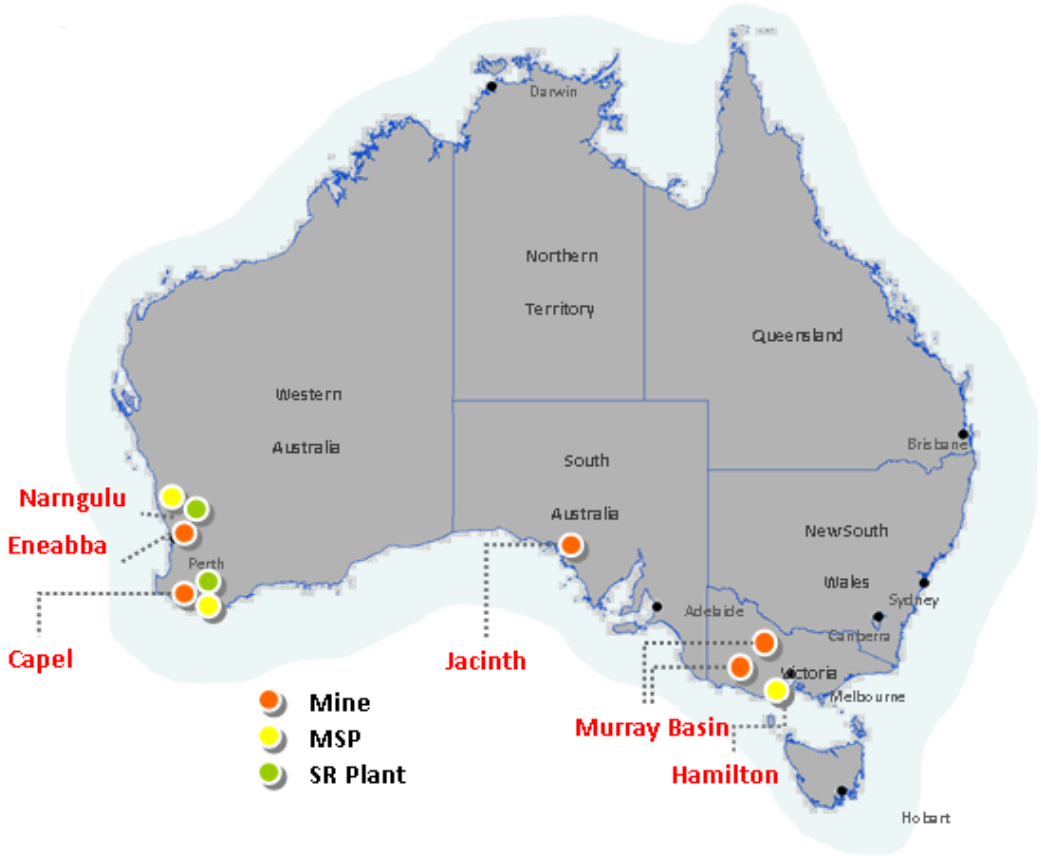
JUNE 2012

Iluka is the largest global producer of synthetic rutile (SR), with a market share of around 50 per cent of the current production output of this product. This paper describes Iluka's SR processes, capabilities and development plans.

Background Information

Iluka has four synthetic rutile plants. Two of these, SR1 and SR2, are located at Capel, approximately 200 kilometres south of Perth in Western Australia. The other two kilns, SR3 and SR4, are located at Narngulu, near Geraldton, Western Australia.

Figure 1 Location of Iluka's Synthetic Rutile Plants



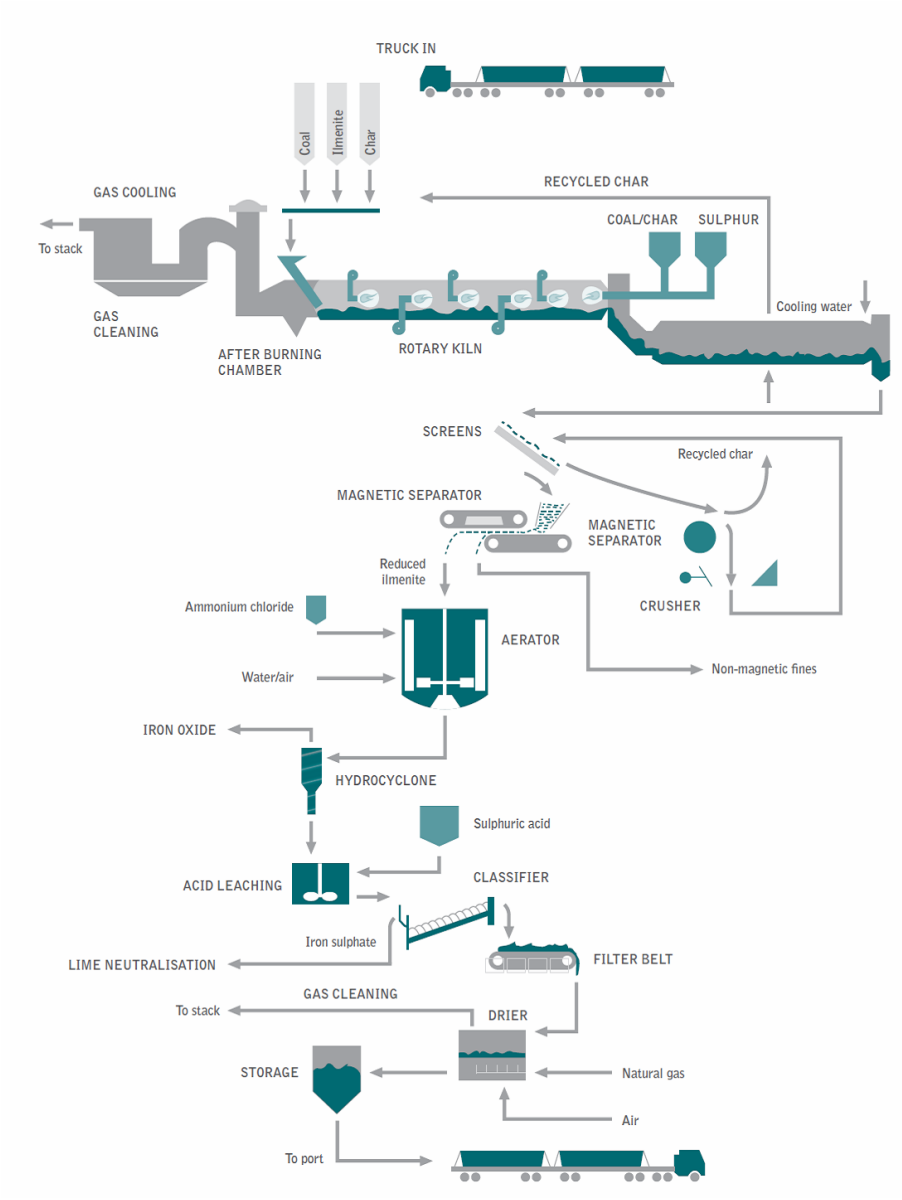
Currently Iluka operates two kilns (SR2 and SR3) but has plans to restart the other two kilns as market demand allows. In 2011, Iluka produced 286,000 tonnes of synthetic rutile and is forecast to produce approximately 310,000 tonnes (kt) in 2012. With all four kilns running, Iluka has the potential to produce between 500kt and 550kt tonnes of SR.

ILUKA'S SYNTHETIC RUTILE PROCESSES

The Basic Process

Synthetic rutile is a chemically modified form of ilmenite, which has had most of the non titanium components removed. The product's composition is between 85 per cent and 95 per cent titanium dioxide (TiO_2). The upper grade TiO_2 products are similar to the naturally-formed mineral – rutile, from which SR derives its name. The ilmenite used in the process generally has between 57 and 60 per cent TiO_2 and yields about 0.6 tonnes of SR for every tonne of ilmenite used. An overview of the SR process is shown in Figure 2. The main objective of the process is to remove the iron component of the ilmenite by reducing it to metal and "leaching" it to leave an upgraded titania product. This process is known as the Becher Process, after its inventor, Dr Robert Becher, who developed the process in Western Australia in 1961. The process was commercialised by Iluka's predecessor company, RGC, in 1969.

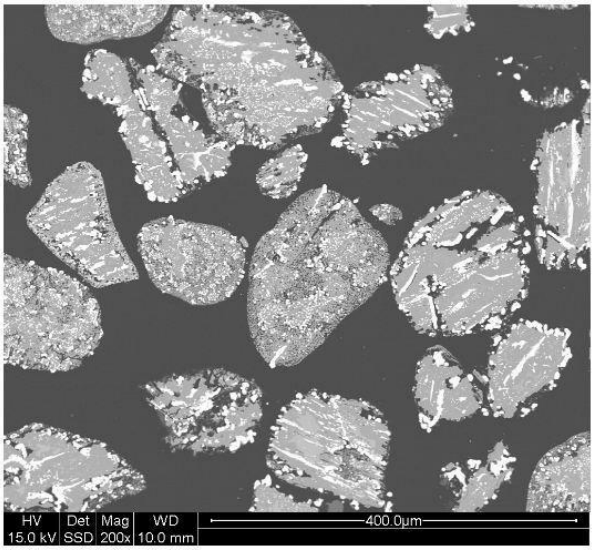
Figure 2 Schematic of the overall SR process



The reduction step involves reacting the ilmenite in a large, continuously fed, rotary kiln such as the one shown in Figure 3. The residence time in the kiln is approximately twelve hours. The heat source is coal, and the source of the chemical reductant (carbon monoxide) is also coal, which is added as lump coal with the ilmenite at the feed-end of the kiln. Controlled air addition allows enough heat to be generated for reduction reactions to occur (~1,100 degrees Celsius), and carbon monoxide to be generated. The carbon monoxide is oxygen deficient, and scavenges some of the oxygen from the ilmenite structure. By this process, most of the iron portion of the ilmenite is converted to metallic iron. The formation of iron particles within the reduced ilmenite grain can be seen in Figure 4.

Figure 3 Rotary kiln used to reduce ilmenite

Figure 4 Photomicrograph of reduced ilmenite

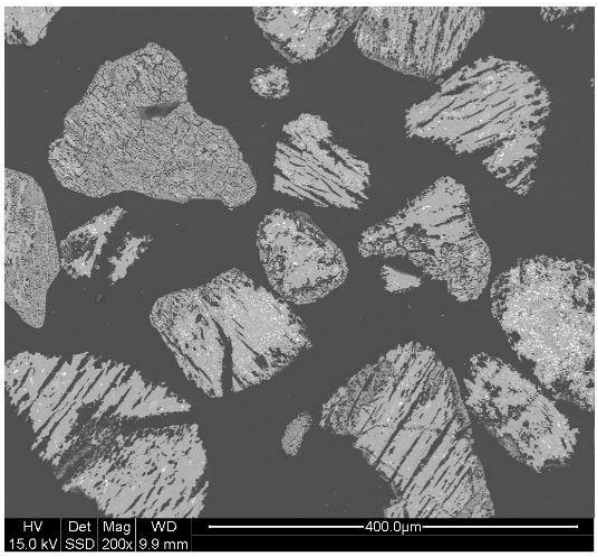


The kiln product is a highly magnetic mineral due to the presence of metallic iron. It is the same size as the original ilmenite and is slightly more porous.

Post reduction and cooling, the reduced ilmenite undergoes screening and magnetic separation to remove partially burnt coal (in the form of char) and some of the other mineral impurities which are non-magnetic.

The product is then aerated, a form of accelerated rusting, which involves the removal of the metallic iron from the reduced ilmenite grain. The reduced ilmenite is mixed in tanks with water and air in a batch process. Ammonium chloride is added as a catalyst to accelerate the process. Preheating of liquor using heat scavenged from elsewhere in the process, is also used to accelerate the process. In these conditions the metallic iron dissolves, migrates through the pores within the particle and precipitates in the bulk solution as very fine iron oxides, 0.1 µm to 10 µm in diameter. After aeration, the sand grains lose up to 40 per cent of their original weight and consist of a porous matrix of titanium dioxide of about 88 per cent purity (Figure 5) but retain their original particle size. Wet separation of the fine iron oxide from the coarse titanium mineral particles is done using hydro-cyclones.

Figure 5 Photomicrograph of SR



The iron oxides are deposited in settling dams, and the ammonium chloride liquor is recovered and recycled.

Synthetic Rutile Products

A number of SR products can be made in the process, depending on the quality of the ilmenite used in the process and the various additional processing steps utilised.

In the Standard SR process, the synthetic rutile is filtered and dried, and is ready for sale. In the Premium SR process, the synthetic rutile is upgraded by leaching with dilute sulphuric acid to remove the iron and manganese phases resulting from sulphur addition to the reduction process and produces a synthetic rutile of 91 per cent to 93 per cent TiO_2 grade. Sulphur addition is made only in the Premium SR process. The sulphur improves iron and manganese removal in the downstream process by reacting in the kiln with the manganese, and stopping the stabilisation of irreducible iron/titanium compounds. After filtering and drying the Premium SR is ready for sale. Overall TiO_2 recovery from ilmenite in the SR process is around 95 per cent.

SREP (Synthetic Rutile Enhanced Product) is a product with even higher TiO_2 and lower impurity levels, in particular radionuclide levels. All ilmenites contain low levels of uranium and thorium, plus their daughter radionuclides. However, in some mineral sands deposits these levels can be elevated to around 500ppm U plus Th. The elements tend to remain with the TiO_2 during the SR process and so are found in even higher concentrations in the SR product. The SREP process adds a boron-based flux during the reduction phase of production. This flux forms a glass phase within the kilns on the surface of the ilmenite grains which attracts the radionuclides from the ilmenite grains during reduction. The glass phase is subsequently removed from SR product be a combination of alkali and acid leaching, thus reducing the U and Th within the SR to sufficiently low levels. Other impurities are also as a consequence of this process and hence the TiO_2 content of SREP is greater than 94 per cent.

In 2011, Iluka added another SR product to its range. SR85 is a product made from Murray Basin ilmenite. Previously, this ilmenite was considered a waste product due to its high impurity levels. However, plant trials during 2011 demonstrated that an 85 per cent TiO_2 product could be made from this ilmenite. The product has gained market acceptance by some customers and is sold under contract to a major pigment producer.

Synthetic Rutile Feedstock

The SR process uses ilmenite as a feedstock. As most of the impurities within the ilmenites end up in the product itself, the process requires ilmenites within a well defined chemical and sizing range in order to make SR products meeting customer requirements. Generally SR ilmenites (as they are termed) need to have TiO_2 contents of between 57 and 62 per cent and ferrous iron levels of less than 15 per cent. Other important impurities are Mn, Mg, Cr, Si, Al, Ca, P, Th and U. Sizing is also important, with customers generally requiring SR products with average grain sizes above 100 microns. Finer grain sizes can be lost in their processes and therefore are seen as suboptimal by some customers.

Iluka sources its SR feedstock predominantly from its Western Australian mines (Eneabba, Tutunup South) or from third party supplies within Western Australia. Smaller volumes are purchased from overseas ilmenite producers. As mentioned above, in 2011 Iluka started to test ilmenites from both South Australia (Eucla Basin) and the Murray Basin in Victoria. In 2012 Iluka plans to test ilmenite produced from its Virginia mine in the United States of America, with the expectation of producing a high TiO_2 product, referred to as SR Ultra.

Energy Efficiencies

The SR process uses large amounts of coal in its process and generates CO_2 emissions. However, the company has taken steps to reduce emissions and conserve energy. A waste heat recovery power generation plant operates at one of the two synthetic rutile kilns (SR2), at Iluka's South West operation in Western Australia. Incorporated into the kiln waste-gas circuit, it recovers the residual energy from the reduction kiln process. Power is produced by an alternator driven by a steam turbine. Steam production is achieved by scavenging waste heat from the kiln off gas using a three-stage counter current heat exchange system. This superheats and pressurises the steam to 490°C and 4,000 kPa. The heat exchange system also has the desired effect of cooling the waste-gas prior to cleaning and exhaust. Approximately 6 MW of electrical energy is produced with SR2 consuming approximately 5 MW, with the excess (approximately 1 MW) exported to the power grid. Iluka obtains partial credits for this power, which offsets power use from other sites.

Synthetic Rutile Markets

Iluka's SR products are sold exclusively as feedstocks into the titanium pigment and titanium metal markets. In the titanium pigment industry, SR is used by those producers utilising the chloride process to make pigment. This process uses chlorine gas plus petroleum coke in a fluid bed reactor to convert the titanium and other metals found in the SR to gaseous metal chlorides. The titanium tetrachloride is purified by a process of distillation and then converted to TiO_2 by burning the chloride gas in oxygen. Chloride pigment producers account for roughly half the global pigment capacity.

The other half is made up of sulphate pigment producers, who use sulphuric acid to dissolve titanium feedstocks and then extract the TiO_2 from solution by hydrolysis. As traditional SR does not dissolve in sulphuric acid, SR is sold exclusively to the chloride pigment producers. Because of this, and its high TiO_2 content, it is often referred to as a "high grade chloride feedstock". However, as outlined below, Iluka is actively developing a SR product which will dissolve in sulphuric acid and therefore will be suitable for the sulphate pigment market.

SR used in the manufacture of titanium metal follows a similar route to that of the chloride pigment process, except that the titanium tetrachloride is reacted with Mg powder to form a titanium sponge plus $MgCl_2$.

The titanium pigment market has grown steadily over the last decade, with the exception of 2009 when the global financial crisis resulted in a reduction in both pigment and metal demand. Since then, demand levels have returned to their pre-2009 growth trends of around four per cent per annum for pigment and over five per cent per annum for titanium metal.

Synthetic Rutile Developments

Since the original commercialisation of the Becher process, Iluka has continuously investigated ways of enhancing the process. The SREP process is an excellent example of the types of development which have been made over the last 40 years.

Since 2009, Iluka's focus on technical and product development has been re-invigorated with the recognition that innovations in these areas will provide Iluka with on-going competitive advantage. The development of new SR products has been central to the overall focus on product and technical development. Iluka's SR development currently deals with the following main areas:

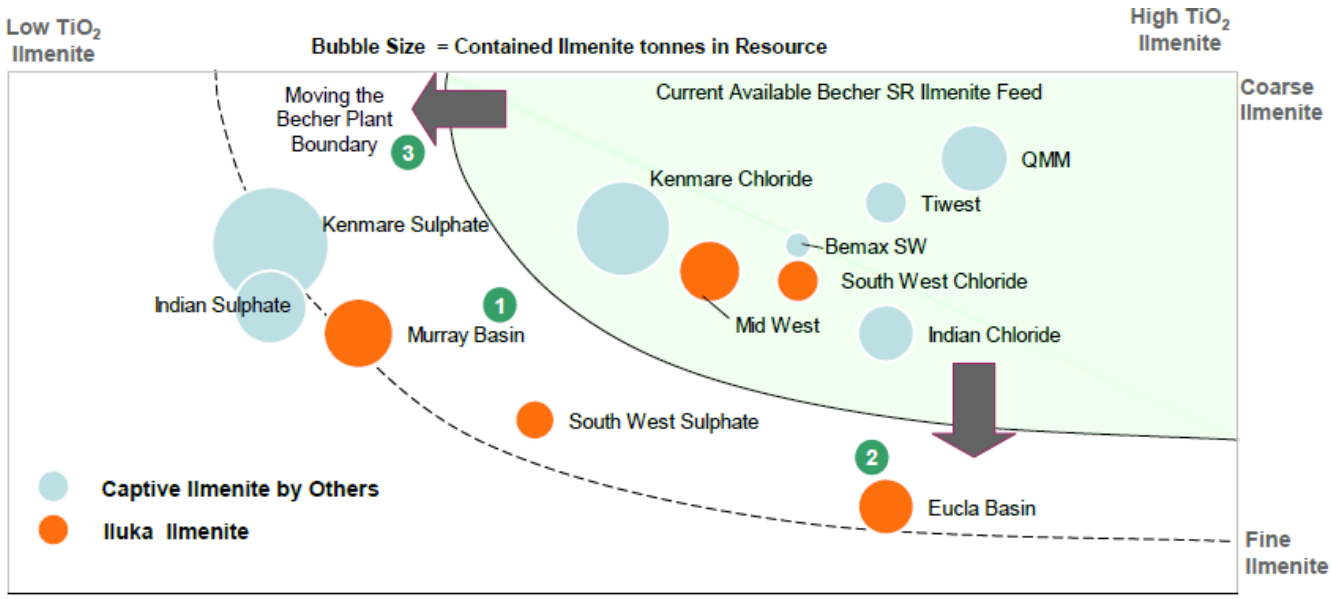
- Using a wider range of ilmenite qualities within the Becher process
- Developing new SR products from these ilmenites
- Developing SR products which can used by the sulphate pigment process
- Developing new ilmenite upgrading processes

These areas of development are outlined in greater detail below.

Using a Wider Range of Ilmenites

Historically, the Becher process has been restricted to using ilmenites with a minimum TiO_2 content of 58 per cent. Impurities such as magnesium, manganese, chromium, silica, aluminium and radionuclides are also important elements to control in the final SR product. As many of these impurities are upgraded in the SR process, their levels in SR ilmenite feedstocks are critical to product quality. Grain size of the SR product is also an important criteria, as fine mineral grains can be lost to the pigment chlorinators before reacting. As such, the ilmenites used in the Becher process have been restricted to those of a specific chemistry and sizing. However, ilmenites found in many mineral sands deposits do not match the criteria needed. Iluka is therefore actively developing methods to allow ilmenites outside the traditional "envelope' to be utilised in the SR process (see Figure 6).

Figure 6 Schematic of wider range of ilmenites used in the SR process



Since 2009, Iluka has successfully incorporated both Murray Basin ilmenite and Eucla Basin ilmenites into its SR feedstock mix. The Murray Basin ilmenite has been used to produce a new product – SR 85, which contains a minimum of 85% TiO_2. Previously this ilmenite was a waste stream from the Hamilton mineral separation plant. SR85 is now sold in commercial quantities under contract. The Eucla Basin ilmenite has been blended into a number of SR products, including Iluka's Premium Grade SR and SREP.

Developing New Synthetic Rutile Products

In addition to the products mentioned above, Iluka is developing SR products with higher TiO_2 contents and lower impurities levels, particularly radionuclide levels. The main feedstock for these products is expected to come from Iluka's Virginia operations in the USA, where the ilmenite mined is of a very high quality. Plant trials using this ilmenite will be conducted during 2012.

Acid Soluble Synthetic Rutile (ASSR)

As mentioned under the marketing section, SR has historically been sold exclusively to those pigment producers using the chloride process. This is because the mineral phases created by the SR process do not dissolve readily in sulphuric acid and therefore are unsuitable for the sulphate pigment process. However, Iluka has been investigating ways of changing the phase chemistry of the SR product, such that the SR is reactive in sulphuric acid and dissolves in this acid. This development product is termed Acid Soluble SR (or ASSR). The product has been successfully produced at a pilot plant level by adjusting a number of the kiln variables. A number of plant trials were conducted in 2011, which provided valuable data to the commercialisation of ASSR. Further trials are planned for 2012 and 2013. Samples of ASSR have been generated and are being evaluated by Iluka's customers.

The advantages of ASSR are that it will potentially use more of the Murray Basin ilmenites, which are currently sold as products in their own right. ASSR will add value to these ilmenites through the production of a higher margin product. The product will also allow Iluka to sell a high grade product into the sulphate pigment market - a sector to which Iluka currently has little exposure.

Developing New Ilmenite Upgrading Processes

Iluka recognises limitations to the Becher SR process. In particular, the process uses a high ratio of coal per tonne of SR and hence has a large carbon footprint. In addition, the process gains no (or very little) value from the iron present in the ilmenite. Until recently, the iron oxide produced as a by-product of the SR process was disposed of in waste dams. Currently, Iluka is able to sell its iron oxide residue as a feedstock to iron producers.

While Iluka intends to first utilise its idle SR capacity before building new ilmenite upgrading capacity, it does recognise that new technologies can provide improved operating cost, lower capital expenditure and lower carbon footprints. The company is therefore actively developing alternatives to the Becher process, which could be implemented post the full utilisation of the four existing Becher plants.

Summary

Iluka has been involved in the production of synthetic rutile for over forty years. The company is the largest global producer of this key high grade feedstock for the chloride pigment market. It operates four kilns, two of which are available for reactivation. The company has made numerous improvements to the process and has progressively optimised the output of its SR plants.

Development of the process has continued in recent years with a renewed focus associated with the establishment of a dedicated Product and Development function, part of the responsibility of which is new product development utilising upgrading facilities. Much of this work has focussed on the use of non-traditional ilmenites as feedstock to the process and the development of a SR product suitable to the sulphate pigment market. This work will continue, while Iluka is also actively investigating newer forms of technology to continue to evolve ilmenite upgrading opportunities.

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