

Fluxes for Soldering

What is a flux?

IPC defines a flux as 'a chemically and physically active formula which promotes wetting of a metal surface by molten solder, by removing the oxide or other surface films from the base metals and the solder. The flux also protects the surfaces from re-oxidation during soldering and alters the surface tension of the molten solder and the base metal.'

Fluxes are used to:

- assist the wetting process by removing oxidised layers from metallic surfaces, and by modifying the surface tensions
- protect the surfaces of both the solder and the parts to be soldered from oxidation during the soldering process
- assist in the transfer of heat between parts being soldered, and thus help equalise their temperatures.

The constituents of flux

A flux may be solid, pasty or liquid, depending on how it is to be used. Its principal constituents are:

- a 'flux base', together with widely variable quantities of
- 'activators' and
- solvents.

When used for wave soldering, fluxes are generally 'low solids' with perhaps only a couple of percent of flux, and the balance solvents. The resulting materials are relatively thin and mobile. However, when formulated as part of a paste for reflow soldering, the flux will also contain additives to improve the paste 'rheology' (flow characteristics) and 'tackiness', and to help slow the sedimentation of solder particles. In many cases, paste components will have more than one function.

For most fluxes:

- the solvent content aids flux (or paste) application, but is volatilised during the pre-heat stages of the soldering process
- the flux base is fluid at soldering temperatures and has good heat transfer properties.

An additional, essential requirement for electronic soldering is that any residues should be solid, relatively inert, and have good insulating properties.

Categorising fluxes

Traditionally there were three main ways of categorising a flux:

1. by its main active ingredients
2. by the type of any solvent used for the removal of flux residues, for example, organic liquids or water
3. by its efficacy as a flux, usually related to its degree of chemical activity, and proportional to its potential to corrode surfaces which it contacts.

In terms of active ingredients, there are two main categories of flux base material for which details are given in the sections which follow:

1. fluxes based on rosin and rosin substitutes
2. water-washable organic (non-rosin) fluxes.

While inorganic fluxes are used for metal-working purposes, they are not generally used for applications in advanced printed circuit assembly because of their potential for corrosion.

Rosin

Most fluxes which are soluble in organic liquids are resins dissolved in a combination of aliphatic alcohols. Most of the resin used is based on colophony (usually called rosin), which is a natural product obtained from the sap tapped from various species of pine tree, steam-distilled to produce liquid turpentine and solid colophony. Each source of rosin has its own chemical make-up and characteristics, and careful selection is needed to ensure even moderate uniformity and repeatability of the resulting flux.

Rosin has been in common use as an electronics flux because it has a combination of favourable properties:

- It is easily available and has well-known chemistry
- At soldering temperature, as a liquid, it wets tarnished metal surfaces and has a sufficiently low viscosity to remove reaction products

- It is active as a flux above 70°C, but below that temperature becomes a varnish with good insulating properties and no corrosivity – a cheap, conformal coat which excludes water and immobilises water already there
- Rosin is soluble in solvents with reasonable/useable vapour pressures. It melts at a sufficiently low temperature, but is reasonably stable at reflow temperatures. It is good at modifying paste rheology and has natural 'tack'.

With its solid state and glassy structure, rosin is not an effective flux until it melts, when the material has sufficient mobility to enter into reactions with the surfaces to be joined. It starts to soften at 50–70°C and is fully fluid at about 120°C, at which temperature plain rosin will clean lightly oxidised copper. However, joint materials are frequently more severely oxidised and, in order to enhance the flux action and give the right results in an acceptable time, rosin is normally accompanied by a chemical system, called an activator.

Flux activators

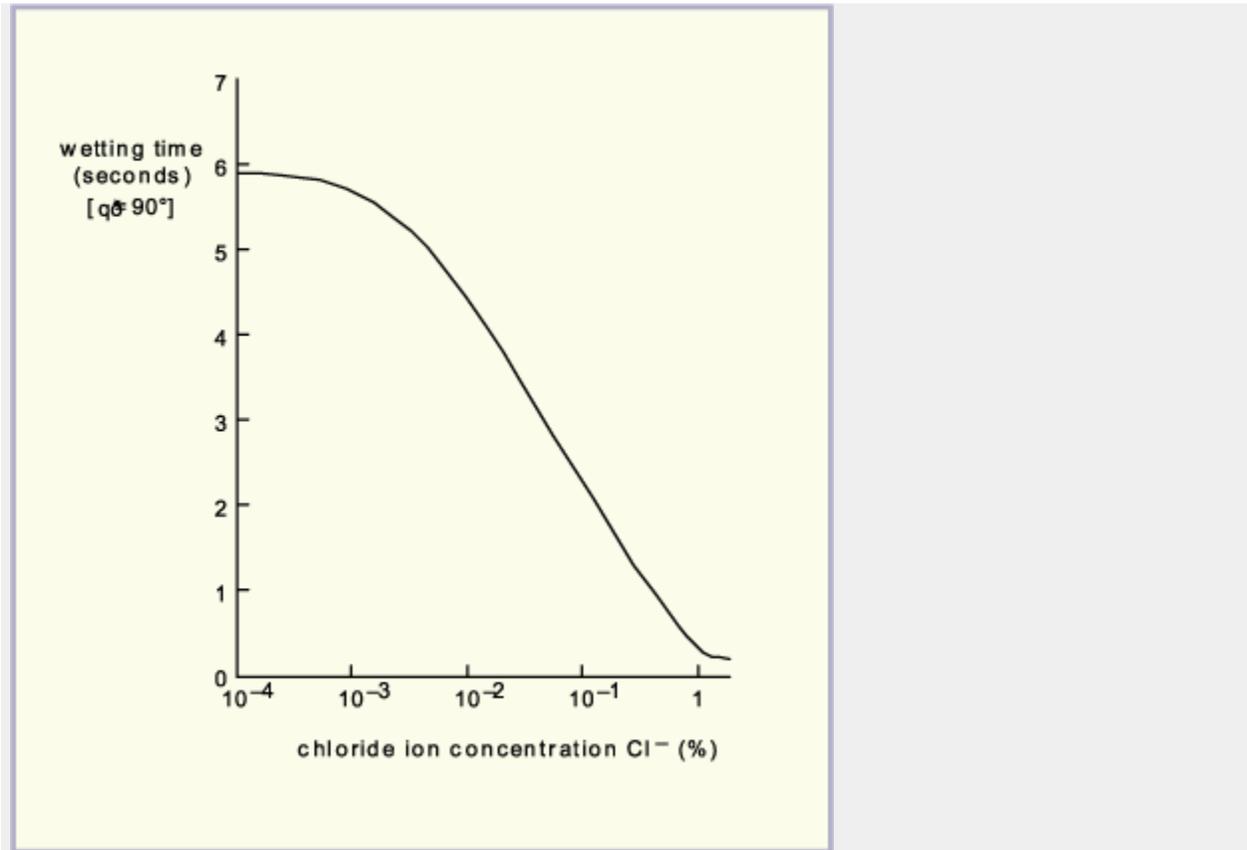
Despite the widely recognised term 'activator', these added chemicals do not activate the rosin, but simply act directly in addition to it. Two salts were commonly used as activators:

- dimethyl ammonium chloride $(\text{CH}_3)_2\text{NH}\cdot\text{HCl}$
- diethyl ammonium chloride $(\text{CH}_3\text{CH}_2)_2\text{NH}\cdot\text{HCl}$

These salts are readily soluble in alcohol or water (though not in colophony itself), and decompose at slightly below the soldering temperature, yielding hydrochloric acid. The 'activity' of this type of flux is expressed as a percentage of chloride ions (Cl^-) relative to the rosin content.

Both activity and corrosivity depend on the percentage of activator. Figure 1 illustrates the effect on the wetting time for clean copper at 235°C of increasing the chloride ion content in rosin flux.

Figure 1: Effect of increasing chloride activator content on the wetting time of clean copper by solder



Organics containing active functional groups such as carboxyl ($-\text{COOH}$) and amine ($-\text{NH}_2$, $-\text{NHR}$, $-\text{NR}_2$) are also good fluxing agents, and are now in more common use because of their reduced corrosivity. However, to achieve the same effectiveness as chloride-containing fluxes, a much larger addition of activator is needed, and hence these 'halide-free' materials are generally used only for mildly activated fluxes.

Rosin flux designations

In the majority of applications pure rosin (what used to be called 'Type R' flux) is insufficiently active to remove surface oxidation and produce good wetting, so activators are added to improve the wetting capability. In many previous specifications, such as the long-established US military QQ-S series of specifications, the level of activation was indicated by the following designations:

- Type RMA (mildly activated rosin) fluxes contain typically 0.2% of mild activators, and are the most commonly used in electronics applications.

- Type RA (activated rosin) fluxes were developed for applications where mildly activated fluxes were too weak, but have residues which are relatively corrosive in humid conditions. Where such 'fully activated' fluxes are used, residues must be thoroughly cleaned, using solvents which will completely remove both polar and non-polar soils. These fluxes are now little used for critical electronic applications.
- Type SRA (superactivated rosin) fluxes have formulations that do not meet even general electronic specifications. They are so strong chemically that they allow soldering to bare Kovar, nickel, and some stainless steels. The flux residues are extremely active, and thorough removal is mandatory.

Rosin substitutes

Colophony is a natural product and hence variable in quality, depending on source and processing. Synthetic resin substitutes have sought to provide a more consistent and purer product. One commercially available substitute is based on the ester pentaerythritol tetrabenzoate: this fumes less and spatters less than rosin fluxes during the soldering operation, and its residues are even less corrosive than those of colophony, and so need not be removed after soldering, unless further activators have been added.

A wide range of other organic materials may be used as fluxes, and specifications often distinguish between fluxes which are primarily resin compositions, or are substantially free of both colophony and resin materials, in which case they are classified as 'organic' types.

'Water soluble' fluxes

This category is based on a solution of strong activators which are designed to produce post-soldering residues which dissolve in water. The activators are generally more aggressive than those found in rosin fluxes, so are better able to deal with poorly solderable surfaces.

However, both the activators themselves and the metal salts produced during the soldering process are potentially more damaging to the circuit and thorough washing is necessary. This is not always easy to achieve with high-density circuits: the difficulty of cleaning under a component increases as the fourth power of the inverse of the stand-off – a 0.1mm gap is 16 times more difficult to clean than a 0.2mm gap!

Flux classification 'by results'

There has always been a confusing mixture of national and international specifications for fluxes, dominated by US military specifications. However, over recent years:

- the US military has begun a process of moving from supporting the preparation and policing of very detailed specifications towards putting the onus on suppliers to prove fitness for purpose
- standards produced by IPC have gained wider acceptance
- the former insistence on cleaning everything regardless has been replaced by an understanding that inefficient cleaning may actually reduce circuit reliability
- there has been a realisation that specifying flux residues is more important than controlling flux constituents, and therefore the emphasis should be on assessing the performance of the flux as well as defining the flux formulation
- The outcome has been a transition towards using the IPC flux specification ANSI/JSTD-004 (formerly IPC-SF-818), and the ANSI/JSTD-005 (formerly IPCSF819) equivalent for pastes.

Flux tests

The descriptions below cover the most commonly used tests, most of which are described both in ANSI/J-STD-004 or ANSI/J-STD-005, and in the IPC-TM-650 Test Methods Manual. The sections of this manual may be freely downloaded at <http://www.ipc.org>: go to 'Online resources' and then 'IPC documents available for download'. Similar tests are to be found in British Standard BS EN ISO 9455.

Flux solids content

IPC-TM-650 Method 2.3.34 is a measure of the material left behind when the flux is heated for one-hour periods at 85°C until a constant weight is obtained.

Halide content

There are two tests used: the silver chromate test (IPC-TM-650 Method 2.3.33) detects halide concentrations above a pre-set level by looking for a colour change in a chemical absorbed on a paper when a drop of flux is placed on it; specific ion titration (IPC-TM-650 Method 2.3.35) gives a

direct measure of halide content, and can detect halide levels as low as 20ppm.

Flux corrosion

In IPC-TM-650 Method 2.6.15, a fixed weight of the non-volatile matter from the flux is added to a fixed weight of 60:40 tin-lead solder in a depression in a sheet of copper. This sheet is floated on a solder bath to reflow the alloy, and then stored at 40°C and 93% RH (relative humidity) for ten days, after which it is inspected for 'signs of corrosion' in or around the flux residues. This test is most severe on low-solids fluxes (which have to be concentrated for the test) and difficult to interpret.

Flux-induced corrosion (copper mirror test)

A measure of the chemical corrosivity of a flux, the copper mirror test is referenced in a number of specifications (such as IPC-TM-650 Method 2.3.32), and is in common use as an indicator of the level of soluble ionic and aggressive activators in a flux. The test assesses the corrosion by cold flux (23°C) in a humid environment (50% RH) of a very thin film of copper on a glass slide: if there is significant corrosion, the film becomes transparent.

For liquid fluxes, the test involves placing two drops of a 35% solution of the flux on a 'copper mirror'. This is a glass microscope slide on which has been deposited approximately 30–50nm of copper, defined as a Cu thickness which allows the transmission $10\pm 5\%$ of normally incident 500nm wavelength light. Typically, a flux made of pure water-white gum rosin and isopropanol acts as a control, and a drop of it is also placed on the mirror. The mirror is then placed in a controlled environment of $23\pm 2^\circ\text{C}$ and $50\pm 5\%$ relative humidity for 24 hours, rinsed in isopropanol and examined for any spots or areas where the copper has been removed by the flux.

The test has several drawbacks which have led critics to question its relevance.

- The test indicates the potential corrosivity of the raw or unheated flux, which is not always indicative of the corrosivity of partially-heated flux (such as flux present on the component side of an assembly during wave soldering) or of fully heated flux (such as flux on the wiring side of a board)
- The results can be difficult to interpret, being somewhat subjective and with no middle ground between pass or fail.

However, the test is quick and easy to perform, and its results correlate well with those of the more lengthy and involved surface insulation resistance test. It is widely accepted and is especially useful as a pre-screening test when testing a new formulation or as a routine incoming materials check.

In some versions of this test, it is applied to solder paste by first extracting any ionic species by boiling a sample of paste in solvent (propan-2-ol) and then concentrating the extract to approximately 35% solids by weight. It has been suggested, correctly, that this procedure is unfairly weighted against pastes with a low-solids flux content, so IPC-TM-650 Method 2.3.32 applies solder paste direct to the mirror.

Surface insulation resistance (SIR testing)

Harmful residues from the flux (or indeed other process chemicals) may provide a conductive path across a circuit. The surface insulation resistance (SIR) test of IPC-TM-650 Method 2.6.3.3 detects the effect of flux residues on the electrical characteristics of conductor patterns. The board is lightly brushed with flux and then floated on the surface of a bath of solder, flux side down. The resistance of the soldered circuit is then checked to be a minimum of 100MW both at the outset and after a period of storage in warm, damp conditions.

SIR is a lengthy but worthwhile flux evaluation test. Of all the test methods available, this one best simulates the real board assembly application. This is because:

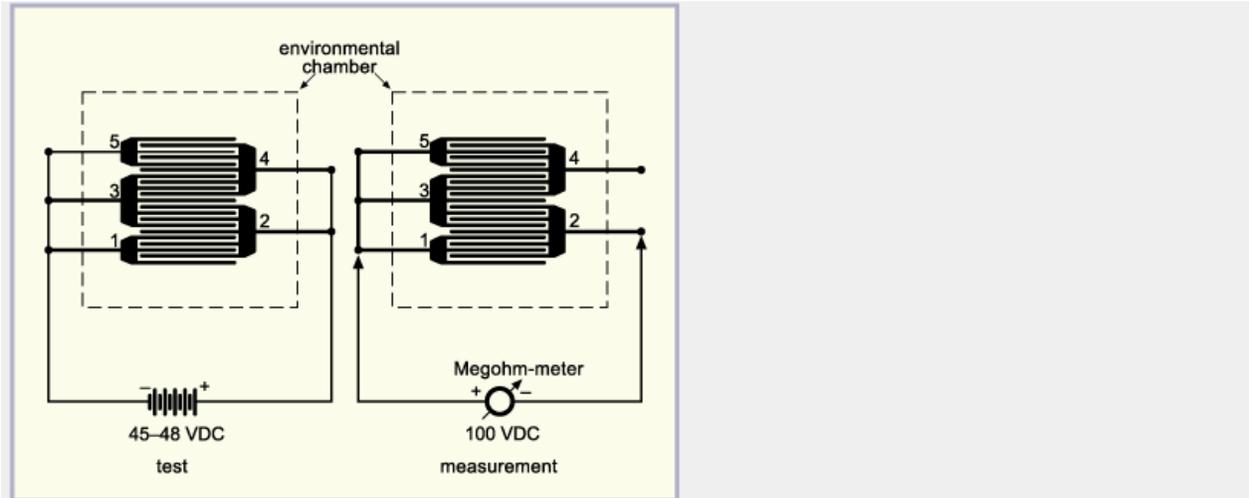
- the soldering process is part of the sample preparation
- the sample substrate is itself a circuit board
- the variable measured, leakage current, is relevant to most circuitry.

Sample preparation simulates the assembly process as closely as possible, including any delays in transfer between processes if this is significant. For wave soldering, preparation includes fluxing the test board and soldering it; for solder paste, this becomes printing and reflow. Cleaning would also be carried out if the process normally includes this. The parts are then subjected to a defined combination of voltage, temperature and humidity determined by the classification of the product and re-tested over an extended period (for example, measuring after one, four and twenty one days).

To maximise the sensitivity of the test, leakage current is measured between long parallel lines at different electrical potentials. Instead of making very long and narrow circuits, electrically equivalent inter-

digitated 'combs' are typically used, as indicated in Figure 2. Traditionally, measurements are reported as insulation resistance (in MW) rather than as leakage current . The diagrams in Figure 2 show typical circuitry used to apply a bias voltage during test and to measure the resistance with a reverse polarity test voltage applied.

Figure 2: SIR testing arrangement



This test is not only used for evaluating fluxes, but is often found as a measure of surface contamination or the purity of surface coatings or encapsulations.

ANSI/J-STD-004 classification scheme

The classification is based on the materials used and the response of the flux to each of the tests above, judged against the requirements of Table 1.

Table 1: Flux activity classification tests					
Flux type	Halide level	Copper mirror	Silver chromate	Corrosion	Conditions for passing 100 MW SIR
L0	0.0%	No evidence of mirror breakthrough	Pass	No evidence of corrosion	Both cleaned and uncleaned
L1	< 0.5%		Pass		
M0	0.0%	Breakthrough in less	Pass	Minor corrosion	Cleaned or

M1	0.5 – 2%	than 50% of test area	Fail	acceptable	uncleaned
H0	0.0%	Breakthrough in more than 50% of test area	Pass	Major corrosion acceptable	Cleaned
H1	> 2%		Fail		

The **first pair of code letters** denotes flux composition, and is chosen from RO (rosin), RE (resin), OR (organic) or IN (inorganic).

The **third code letter** shows flux/flux residue activity, and is chosen from L (low), M (moderate) or H (high).

The **final code character** indicates whether the flux contains halide (1) or is halide-free (0).

Examples of these codes are given in Table 2¹.

¹ Note that there are substantial differences between the ANSI/J-STD-004 classification and that used previously in IPC-SF-818, which did not always specify the material type but included a usage classification.

Table 2: Examples of flux type designations	
ROL0	Rosin-based flux with no halide content, and low flux or flux residue activity. Passes the SIR requirements even when not cleaned.
REM1	Rosin-based flux with 0.5–2.0% halide, and moderate flux or flux residue activity. Depending on the specification, may or may not need to be cleaned in order to pass the SIR requirements.
ORH1	Organic-based flux with >2.0% halide, and high flux or flux residue activity. Must be cleaned in order to pass the SIR requirements.

ANSI/J-STD-004 offers a guideline table (Table 3) which illustrates ‘an appropriate, though not necessarily always accurate, analogy between L, M and H type fluxes with the traditional classes of rosin-based fluxes’.

Table 3: Approximate equivalents for ANSI/J-STD-004 flux categories	
L0 type fluxes	All R, some RMA, some low solids no-clean

L1 type fluxes	Most RMA, some RA
M0 type fluxes	Some RA, some solids no-clean
M1 type fluxes	Most RA, some RSA
H0 type fluxes	Some water-soluble
H1 type fluxes	Some RSA, most water-soluble and synthetic activated

It must be stressed, however, that there is no direct correlation between the ANSI/J-STD-004 designation of a flux, based on results, and any designation based purely on materials. Some fluxes which were formerly considered to be potentially corrosive can now be used; others believed 'safe' have had doubt cast on their long-term reliability.

Source: http://www.ami.ac.uk/courses/topics/0129_ffs/index.html