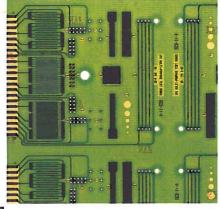
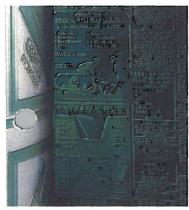
Photoinitiators for UV Curing







A Formulator's Guide

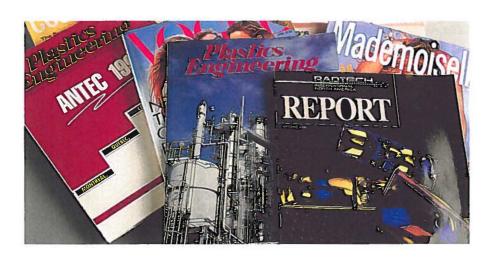


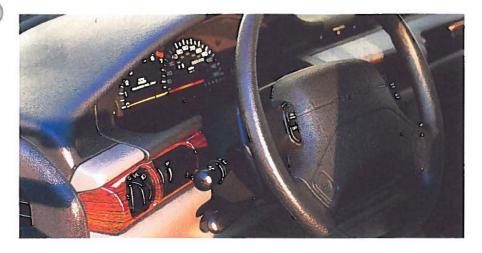


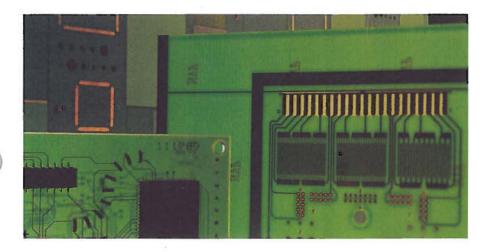
Ciba Additives for Ultraviolet (UV) Curing

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A single technology—UV curing—is used to improve the quality of finished products, at the same time boosting productivity by saving energy, space, time and labor. Also of prime importance is the ability to formulate systems with low, or no volatile organic compounds (VOC), by using UV curing.

In wood, plastic, paper, and other coatings, it has been used to increase line speeds and develop coatings with superior environmental resistance and better gloss.

In screen printing, lithographic printing and flexographic printing, UV curing technology has resulted in major energy and space savings, while deriving the environmental benefit of low VOCs.

Laminating, pressure sensitive and structural adhesives utilizing UV curing exhibit superior chemical and physical properties, while benefitting from the technology's inherent efficiencies.

Printing plates, printed circuit boards, fiber optics, tooling and medical applications are a few additional areas where UV curing has provided a unique combination of benefits. Today, printing plates are produced faster, with far better quality than was previously possible. Printed circuit boards have higher circuit densities and stereolithography has opened up new vistas in prototyping and prosthetics.

Ciba Additives has been involved in the industry since the 1970s, when we introduced Irgacure® 651. Over the past 20 years, we have continued to focus our research on developing photoinitiators to meet evolving customer and industry needs. We have the broadest range of photoinitiators available in the industry. Irgacure and Darocur® photoinitiators cover a wide UV and visible light absorption range. They can be used to give balanced surface and through cure characteristics in clear and pigmented coatings. Ciba Additives research continues to work on developing new answers to customer photoinitiator requirements.

This brochure will explain photoinitiator technology—how and why it developed—and outline the features and benefits of each product. It will help you make an informed decision as to which initiator will help you optimize your formulation.

What is UV Curing?

Some three decades ago, a new process was introduced for curing coatings. The process—ultraviolet light (UV) curing—was developed for the printing, packaging, electronic and wood product industries. Thirty years of research and development in both application specific products and radiation curing equipment have led the UV curing industry to its present status. The industry is poised and well positioned to embark on its most ambitious growth period to date. Today, formulators have an array of choices from advanced raw materials through application equipment and innovative energy sources.

Industry analysts predict that the UV curing market-with sales of formulated products of greater than \$600 million in the mid-'90s-will achieve sales growth in the 10-15 percent range throughout the remainder of this decade. What makes the UV curing process so attractive? This growth is fueled by the adoption of UV technology by new markets and the expansion of this technology in existing markets. UV curing represents, in a single process, the best non-polluting method of providing manufacturers with a finished product of exceptional beauty, strength and value.

Why Choose UV Curing?

Ultraviolet (UV) curing was first commercially successful in the wood and furniture industries. Today, UV curing is used in a diverse number of industries, including coatings, inks, electronics and adhesives. The UV curing process offers finished product manufacturers at least three outstanding benefits:

- Environmental advantages
- Unique physical properties
- Production efficiencies

Environmental Advantages

UV cured coatings, inks and adhesives have always contained low levels of volatile organic compounds. As we approach the 21st century, such low VOC products become increasingly important, offering manufacturers a way to help in meeting or exceeding current and pending environmental regulations to limit emissions and control air quality. These regulations include the Clean Air Act of 1990, as well as newly developed Environmental Protection Agency (EPA) control technology guidelines that will limit VOCs for a number of processes including coatings, inks and adhesives.



UV curing provides the type of "enhancement technology" that allows manufacturers to make their products more attractive and durable. Finished products exhibit exceptional stain, abrasion and solvent resistance, coupled with superior toughness. Moreover, UV curing allows the printing and ink industries to achieve the highest gloss attainable by any coating method.

Production Efficiencies

Because UV curing is so different from traditional finishing processes, it affords manufacturers a new slate of production opportunities: specialty products can now be made to meet market specific needs, such as optical fibers, photoimaging of printing plates, and fast-curing wood fillers. Moreover, the development of specialized UV lamp systems allows for three dimensional curing, opening markets and uses, including bathroom vanities, molded plastic parts and furniture case goods. Finally, and perhaps most important, the UV curing process greatly reduces drying times and improves production efficiency and provides pollution abatement.



What is the UV Curing Process?

The UV curing process requires a UV lamp which directs UV light onto the formulated product. Photoinitiators absorb the UV energy from the light source, setting in motion a chemical reaction that quickly—in most cases the time is measured in fractions of a second—converts the liquid into a solid, cured film.

The bulk of the formulation contains monomers and oligomers. Monomers are low molecular weight materials. They can be monofunctional, bifunctional, or multifunctional molecules. These molecules become part of the polymer matrix in the cured coating because of their reactive functional groups. Furthermore, monomers also function as diluents in the formulations. Therefore, monomers are sometimes referred to as reactive diluents.

Oligomers, on the other hand, are high molecular weight viscous materials. The molecular weight ranges from several hundred to several thousand or even higher. Usually the type of oligomer backbone determines the final properties of the coating such as flexibility, toughness, etc. These backbones can be polyether, polyester, polyurethane or other types. The functional groups that provide the linkage between molecules are usually located at both ends of the oligomer molecules. The most common functionality used is the acrylate functional group.

A Photoinitiator can be categorized in different ways:

According to its mechanism:

- •free radical
- •cationic

According to its form:

- •liquid
- ·solid

According to its absorption:

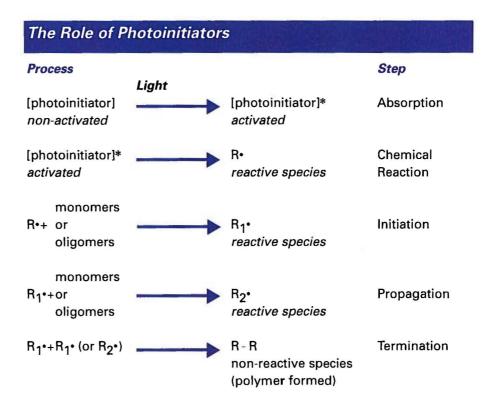
- UV light
- visible light

According to its application:

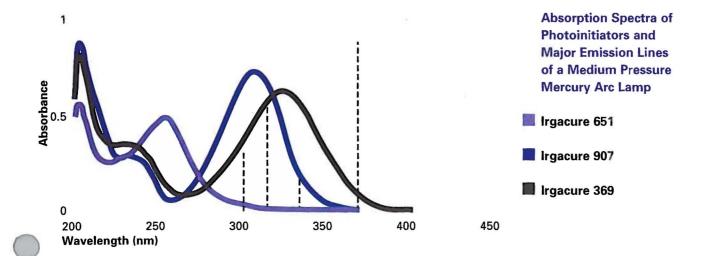
- •clear
- pigmented
- white

The photoinitiator is a critical component of the UV curing process. It is the additive that initiates the polymerization process to quickly reach the final crosslinked product. As UV light energy is emitted, it is absorbed by the photoinitiator in the liquid, causing it to fragment into reactive species. These species can be either free radical or cationic. The majority of systems are based on free radicals which react with the unsaturated compounds in the liquid, and cause them to polymerize. This reaction is almost instantaneous, and is shown below.

Of equal importance to the photoinitiator, however, is the UV light source. Basically, two types of light sources are available: arc light and laser light. The arc type light includes the medium pressure mercury lamp and the high pressure xenon lamp. The medium pressure mercury lamp is currently the overwhelming industry choice because of its high power (200-700 watts/inch) and important emission lines which are absorbed by most commercially available photoinitiators. Regardless of the type of light source, however, the emission spectra of the lamp must overlap the absorption spectrum of the chosen initiator (Figure 1).



Types of Photoinitiators



The free radical class of initiators represents greater than 90% of the commercially used initiator chemistry, while cationic curing has been more limited in scope. Free radical initiators are most typically used with acrylate/methacrylate functional resins and can also be used with unsaturated polyester resins. Cationic curable systems are dependent on the use of epoxy or vinyl ether functional resins.

Free radical initiators can be described as either "hydrogen abstraction type" or "alpha cleavage type." While hydrogen abstraction initiators have their specific uses, especially in three or four-way photoinitiator blends, alpha cleavage initiators are the most widely used. The alpha cleavage initiators have a generally higher efficiency due to their generation of free radicals via a unimolecular process: alpha cleavage initiators need only to absorb light in order to generate radicals; hydrogen abstraction initiators, on the other hand, require an extra step: after absorbing light, the excited state photoinitiator must find a hydrogen donating source in order to generate the free radicals. It is a bimolecular process.

Beyond the obvious differences in chemical structure, the alpha cleavage initiators are typically differentiated by their absorption profiles. A formulator should be interested in two different characteristics of the photoinitiator's absorption curve. First is determining which wavelengths of light are absorbed, and second is the strength of this absorption (molar extinction coefficients). Photoinitiators developed for curing of pigmented films typically have higher molar extinction coefficients between the wavelengths—from 330 nm to 400 nm—than those useful for curing of clear formulations.

Ciba Photoinitiators for UV Curing Overview

In the late 1970s, Ciba Additives addressed the industry need for a photoinitiator with a greatly improved shelf life and superior through cure. Our research and development yielded Irgacure® 651, benzil dimethyl ketal, to replace benzoin ethers. High photospeed combined with improved secondary properties helped make it the initiator of choice for many applications, including coatings on wood, metal and plastic.

In the early 1980s, market demand for a non-yellowing initiator spurred our development of Irgacure 184, a cyclohexyl phenyl ketone. It is an alpha cleavage type photoinitiator. Irgacure 184 combines the characteristics of high photospeed and exceptional storage life with non-yellowing properties, making it an ideal choice for clear coatings on paper, flooring and plastics.

At the same time, the offset and screen ink printing industries needed an initiator that could handle their ever increasing line speeds. Also required was an initiator with improved absorption characteristics, enabling it to compete effectively with other light absorbing components, such as pigments. Our research resulted in a new class of alpha cleavage initiators based on morpholino substituted acetophenones. First offered was Irgacure 907, which provides strong absorption with efficient sensitization by thioxanthones. These properties have made Irgacure 907 a highly efficient initiator for the printing ink and photoresist/solder mask markets.

Irgacure 369, another photoinitiator of the amine substituted acetophenone class, was developed to allow coatings formulators greater flexibility in manufacturing coatings for the graphic arts and electronic industries. Irgacure 369 provides the strong absorption needed for pigmented systems, and has its absorption maximum red-shifted to make more efficient use of the 365 nm emission line from the mercury vapor lamp.

In the mid-1980s Ciba Additives introduced its first liquid photoinitiator, Irgacure 500. It is a mixture

of two initiators: 1-hydroxycyclohexyl phenyl ketone (HCPK) and benzophenone (BP). Irgacure 500 provides an excellent balance of surface and through cure properties and assists curing conducted in air, with no nitrogen blanket required. Irgacure 500 functions by a unique dual photocuring mechanism: the HCPK initiates via an alpha cleavage process, while the BP reacts by a hydrogen abstraction process. Irgacure 2959 is a hydroxy functional initiator providing enhanced miscibility in water-borne systems.

We also acquired the Darocur® line of photoinitiators, all with faint or low odor characteristics, making them ideal candidates for the workplace. Darocur 1173 and Darocur 4265 are liquid photoinitiators. Darocur 1173 is also useful in UV cured coatings that require minimal yellowing.

Darocur 4265, a phosphine oxidebased initiator, was developed to meet the needs of curing highly pigmented white systems. Part of this efficient through cure of whites is due to its photobleaching properties. The phosphine oxide initiator class was extended with the introduction of Irgacure 1700, which is characterized by its stronger absorption beyond 380 nm and an increased yield of free radicals due to the novel molecular structure of the bisacyl phosphine oxide component.

Irgacure 784 DC addresses the market needs for an initiator that responds to visible light sources. Its absorption, even beyond 500 nm, makes it useful for imaging with the 488 nm line of an argon laser. Irgacure 784 DC also makes it possible to develop formulations that will respond to sunlight as well as incandescent or fluorescent light sources.

Irgacure 261 represents a separate class of initiators. It is a cationic initiator, useful for photopolymerizing epoxy resins. It shows most promise in the curing of thick films or pigmented coatings, where its long wavelength absorption is most needed.

A comprehensive listing and description of Ciba photoinitiators follows. Reference charts on page 13 also cross reference initiators by application area.

For additional information about individual products, please read the product bulletins on each of our products, or contact your local Technical Representative (see pg. 44).

Ciba Additives



Ciba Additives is a worldwide technical leader in the development and manufacture of high quality additives for coatings, inks and adhesives. Our mission is to provide innovative chemistry and services for our customers. For more than a quarter of a century, our goal has been to develop materials that help improve the performance and appearance of our customers' products.

We are determined to maintain our leadership in the chemical industry by meeting and anticipating our customers' needs for advanced products and services. Moreover, we are committed to continued innovation in the chemical industry by fostering partnerships with our customers to promote the proper use and disposal of our products. As we turn scientific discoveries into customer-preferred products, we simultaneously pledge to respect the environment, strive to reduce waste at its source and use natural resources wisely.

Our commitment to respect the environment is underscored in one way by our development of photoinitiators for ultraviolet cured coatings. UV cured coatings can enable manufacturers to meet—indeed exceed—the demanding government restrictions on volatile solvent emissions as well as the new limitations on wastewater effluents and solid and liquid waste disposal.

Ciba Additives continues to focus the quality of its products and services. Our businesses in the U.S., Switzerland, Italy and Germany have received ISO 9001 certification. This certification assures our customers that Ciba Additives has met standards in research and development, purchasing, manufacturing, marketing, sales, inspection, shipping, delivery and services. ISO certification is one milestone in a process of continuous improvement.

Our research efforts are fueled by evolving customer needs for improved productivity and product performance, and increasingly stringent environmental demands. Ciba Additives produces and markets a broad range of materials to meet the current and anticipated future needs of our customers. In addition to state-of-the-art photoinitiators for UV curing, we offer formulators a variety of light stabilizers, antioxidants, corrosion inhibitors, and optical brighteners to improve and prolong product performance.

Beyond our considerable investments in research, manufacturing and marketing, Ciba Additives also maintains a strong commitment to environmental and worker safety. As a conscientious member of the Chemical Manufacturers Association (CMA), we have implemented an ambitious program to educate employees and customers about the six Responsible Care® codes of management.

Chronology of Ciba Photoinitiators

Product	Year Introduced	Description
Irgacure 651	1977	General purpose initiator; useful for curing unsaturated polyester resins.
Irgacure 184	1980	Best for non-yellowing applications; also relatively low odor.
Darocur 1173	1980	Liquid non-yellowing photoinitiator. Good solvency properties makes it ideal for making photoinitiator blends.
Irgacure 500	1984	Liquid blend of Irgacure 184 and benzophenone gives good balance of surface cure and through cure.
Irgacure 907	1985	Strong absorption characteristics make it useful for curing pigmented inks and coatings; excellent surface cure initiator.
Darocur 4265	1988	Excellent for curing white inks and coatings.
Irgacure 2959	1988	Very low odor and low volatility photoinitiator; also has terminal hydroxyl group which may be reacted into polymer backbone.
Irgacure 369	1990	Strong broad absorption characteristics make it most useful for curing thick pigmented formulations.
Irgacure 261	1992	Cationic initiator for curing of epoxy resins; useful for thick film or pigmented coating applications.
Irgacure 1700	1994	Most efficient initiator available for curing inks or coatings containing ${\rm TiO_2}$.
Irgacure 784 DC	1994	Visible light initiator responds to all UV and visible light sources including visible lasers.

Selection Criteria

The following chart is designed to assist a formulator in choosing which photoinitiators should be tested in a given type of application:

Ciba Photoinitiator Selection Guide

White

Irgacure 184 Irgacure 500 Darocur 1173	Irgacure 1700 Darocur 4265	Irgacure 907 Irgacure 369	Irgacure 261	Irgacure 784 DC	Primary
1700	Inma 404	1		I 4700	100

Pigmented

Irgacure 1700
Irgacure 2959
Irgacure 651
Darocur 4265

Clear

Irgacure 184
Darocur 1173
Irgacure 651
Irgacure 907
Irgacure 500

Irgacure 184
Irgacure 651
Darocur 1173
Irgacure 1700
Darocur 4265
Irgacure 500

Irgacure 1700 Irgacure 369

Visible

Secondary

Note: (1) Addition of BP/3° amine is recommended if tackfree surface is difficult to achieve in a thin coating. (2) For thick coatings, addition of Irgacure 1700 or Darocur 4265 is suggested. (3) Darocur 1173 and Irgacure 2959 are recommended for water-borne systems. (4) To achieve low odor or volatility, Irgacure 2959 is recommended. (5) Irgacure 184 gives

the best long-term

mance in coatings.

non-yellowing perfor-

Note:

(1) If TiO₂ content is higher than 15%, Irgacure 1700 is recommended.
(2) Addition of Isopropyl Thioxanthone (ITX) is recommended when Irgacure 907 is used.
(3) A low concentration of Irgacure 907 is recommended to minimize yellowing.

Note:

Irgacure 907/ITX is recommended. The ratio ranges from 10/1 to 10/5 in most applications. (2) Addition of Irgacure 184 or Darocur 1173 is recommended along with Irgacure 369 to balance surface and body cure. (3) Irgacure 369 is recommended if pigment content and/or film thickness are high. (4) The combination of a primary and secondary choice is suggested to achieve the best cost/per-

formance formulations.

(1) The combination of

Note:

Cationic

(1) Addition of ITX/Cumene Hydro Peroxide is recommended. (2) Post-thermal treatment is recommended to enhance performance. (3) Cycloaliphatic epoxy resin is suggested for use. (4) The molecular weight and the type of polyols can affect Irgacure 261 performance. (5) Try to avoid nucleophiles in formulations. (6) Irgacure 261 is not recommended for non-yellowing or fast cure coating systems. (7) Irgacure 261 exhibits better performance in thick coatings than in thin coatings.

Note:

light.
(2) Less than 1.0% concentration is usually recommended for most applications; however, the concentration can be as high as 3% when the coating thickness is less than 5 microns.

(1) Irgacure 784 DC has

to be handled under red

Incorporation Methods

When incorporating photoinitiators, it is generally recommended to dissolve the photoinitiator in a monomer and then add the solution to the resin to ensure complete dissolution. A very fine dispersion of photoinitiator in the formulation instead of a fully solubilized material will reduce the probability of light absorption. Liquid photoinitiators can be added to either the monomer or the formulated coating as they are easier to disperse.

Formulating Suggestions

Test Several Initiators - Our selection guide on page 13 was developed to assist the formulator in readily selecting an initiator which is most useful for the application. Because of differences in resin chemistry, lamp configuration, and the desired end properties, it is impossible to know which initiator will be most useful under any given condition. We therefore suggest that two or three different initiators be tested for initial screening. This is particularly important when starting a new formulation or curing under a new set of conditions.

Run a Ladder Study - When selecting a photoinitiator for a new application, it is difficult to know the optimum concentration without conducting a detailed ladder study. The optimum concentration may also be dependent on which is most important, cost efficiency or performance factors. A ladder study covering a range of three or four concentrations is suggested.

Ensure Complete Solubility of the Initiators - Photoinitiators perform optimally only when they have the maximum opportunity to absorb light from the exposure source. To maximize the absorption possibilities, full solution of the initiators in the resin system is mandatory. While all of Ciba's liquid photoinitiators are easily mixed into a wide range of resin systems, solid initiators may require a moderate mixing cycle. In clear coatings, full solubility is easily determined by looking for clarity of the solution. In pigmented formulations, it is advisable to first dissolve the solid initiators in the monomer/ oligomer blend prior to incorporation of the pigments.

Optimum performance properties as well as cost effectiveness is often optimized by testing blends of initiators. Common combinations include the primary initiator along with a sensitizer (Irgacure 907 and Irgacure 339 can be sensitized by ITX), use of a surface cure initiator along with a through cure initiator (e.g., Irgacure 500 for surface cure/Irgacure 184 for through cure), and use of different initiaters to optimize the use of all available light energy (pick one initiator with good short wavelength absorption and another with good long wavelength absorption).

- As your development program proceeds, you are likely to be changing other parameters such as line speed, film thickness, or even the light source. When any of these changes occurs, the concentration of photoinitiator originally selected may not be the best. Reoptimize!

See - UV curable coatings and inks for exterior applications will likely require the presence of stabilizers to prevent degradation due to sunlight. Ciba has developed two different types of light stabilizers: ultraviolet absorbers and hindered amine light stabilizers (HALS). As the HALS do not absorb UV light, they have little or no impact on the cure properties of the photopolymers. Ultraviolet absorbers, however, compete with the photoinitiator for the available light and therefore slow down the cure speed of most formulations. In this case a formulator has two choices. The first is to use a higher concentration of photoinitiator to overcome the screening effect of the ultraviolet absorbers. The second approach is to use an initiator with good long wavelength absorption outside the absorption region of the UV absorbers (Irgacure 369 or Irgacure 1700 are particularly recommended).

generally indicate that the formulation is not able to overcome the oxygen inhibition which is inherent in the UV curing of acrylates. Higher photoinitiator concentrations are generally recommended to overcome this problem. You may also want to concentrate on initiators with proven good surface cure properties, such as Irgacure 907, Irgacure 184 or Irgacure 500.

lems may show up as surface wrinkling or may be manifested through poor performance properties such as adhesion. These problems can often be improved upon by using lower levels of photoinitiators (allowing more light to penetrate through the coating) or by using initiators with better long wave length absorption. Irgacure 369 and Irgacure 1700 are particularly recommended for through cure.

In Summary

Ciba Additives continually strives to produce high quality products which meet customer, manufacturing and environmental demands. We invite you to call us to discuss your UV curing needs. Our technical staff is ready and willing to work with you in designing a photocuring system to meet

your requirements. Further, our staff is always available to answer specific questions on Ciba photoinitiators and other additive combinations.

Table 1Extinction Coefficients of Ciba Photoinitiators at Different Wavelengths Important to Mercury Vapor Lamps

Wave-	Irgacure	Irgacure	Irgacure	Irgacure	Irgacure	Irgacure	Irgacure	Darocur	Irgacure	Irgacure	Darocur
length	184	261	369	500	651	784	907	1173	1700	2959	4265
(nm)	3.177	9.828	1.303	6.635	4.579	7.488	8.857	3.705	3.075	5.032	1.446
254	x 10 ⁴	x 10 ³	x 10 ⁴	x 10 ⁴	x 10 ⁴	x 10 ⁵	x 10 ⁴	x 10 ⁴	x 10 ⁴	x 10 ⁴	× 10 ⁴
302	1.255	8.285	4.387	1.820	1.696	1.940	1.404	1.640	3.198	1.156	4.925
	x 104	x 10 ²	x 10 ⁴	x 10 ³	x 10 ³	x 10 ⁴	x 10 ⁵	x 10 ³	x 10 ³	x 104	x 10 ³
313	1.485	5.470	5.693	1.075	8.500	1.424	1.287	1.015	2.355	3.790	3.752
	x 10 ³	x 10 ²	x 10 ⁴	x 10 ³	x 10 ²	x 10 ⁴	x 10⁵	x 10 ³	x 10 ³	x 10 ⁴	x 10 ³
365	1.350	1.455	7.369	1.650	4.240	2.612	8.240	1.600	6.325	3.300	8.300
	x 10 ²	x 10 ²	x 10 ³	x 10 ²	x 10 ²	x 10 ³	x 10 ³	x 10 ³	x 10 ³	x 10 ²	x 10 ²
405		1.725 x 10 ²				1.197 x 10 ⁵					
435						1.124 x 10 ³					

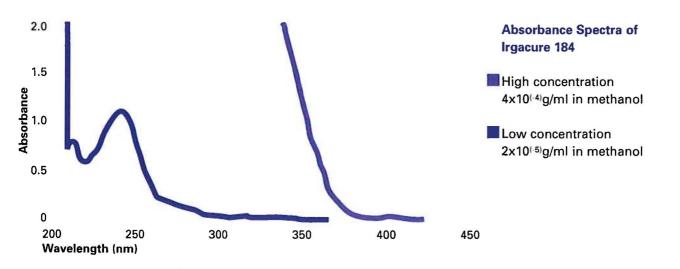
Note: Extinction coefficient is calculated in MeOH solution at room temperature. Its unit is ml gram-1 cm⁻¹.

Irgacure 184 is a highly efficient photoinitiator developed for UV curing coatings, inks and adhesives. It is strongly recommended when non-yellowing properties are critical. Results obtained from an outdoor Florida exposure study show that Irgacure 184 gives better long-term non-yellowing performance than Darocur 1173. Furthermore, Irgacure 184 exhibits lower sensitivity to air inhibition than Darocur 1173 during curing.

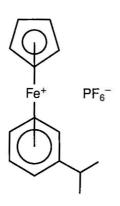
Chemical Structure/Name

1-hydroxycyclohexyl phenyl ketone (HCPK) $C_{13}H_{16}O_2$

Appearance	White granular powder
Molecular weight, g/mole	204.27
Melting range, °C	44-49
Density, g/ml	1.17
UV absorption peaks, nm	240-250
	325-330



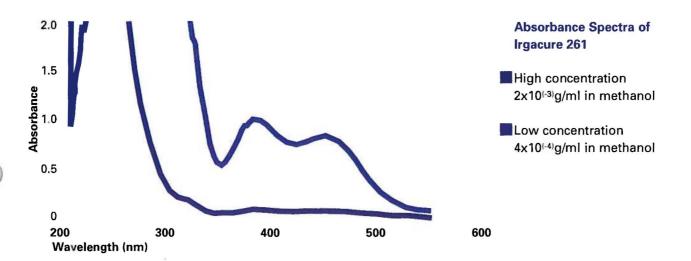
Irgacure 261 is a cationic photoinitiator for epoxy-based applications. The best curing results can be achieved by adding oxidizing agents (i.e., cumene hydroperoxide) and sensitizers (i.e. Isopropyl Thioxanthone) in formulations. In general, Irgacure 261 exhibits better performance in thick coatings than in thin coatings.



Chemical Structure/Name

(η⁵-2,4-cyclopentadien-1-yl)[(1,2,3,4,5,6-η)-(1-methyl ethyl)benzene]-iron(+)-hexafluorophosphate(-1) $C_{14}H_{17}F_6PFe$

Appearance	Yellow powde
Molecular weight, g/mole	386.1
Melting range, °C	85-88
UV absorption peaks, nm	240-250
	395-400
	530-540



Irgacure 369 is an excellent alphaamino acetophenone photoinitiator for non-white pigmented systems. It has strong absorption where several important emission wavelengths from mercury vapor lamps are available. Its absorption maximum is red-shifted compared to the absorption peak of Irgacure 907. Consequently, Irgacure 369 tends to show better through cure than Irgacure 907 in dark color pigmented systems. Irgacure 369 is one of the photoinitiators that exhibits low volatility. No weight loss was detected according to ASTM Test Method D-23369-87.

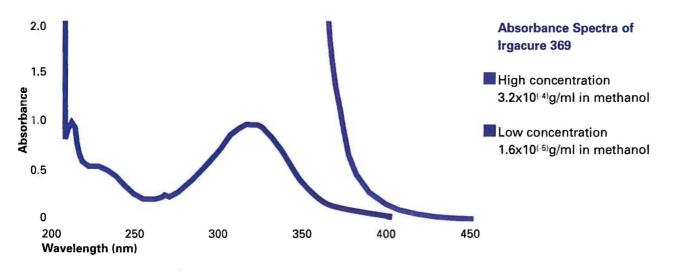
$$\begin{array}{c|c} O & C_2H_5 & CH_3 \\ \hline O & C & C-N & CH_3 \\ \hline CH_2 & CH_3 \end{array}$$

Chemical Structure/Name

2-benzyl-2-N,N-dimethylamino-1-(4-morpholinophenyl)-1-butanone (DBMP)

 $C_{23}H_{28}O_2N_2$

Appearance	Slightly yellow powder
Molecular weight, g/mole	366.5
Melting range, °C	110-114
Density, g/ml	1.18
UV absorption peaks, nm	325-335



Irgacure 500 is the first liquid photoinitiator introduced by Ciba Additives. Its unique composition provides an excellent balance of surface and through cure for clear coatings. Irgacure 500 shows less yellowing in coatings compared to Irgacure 651.

1-Hydroxy cyclohexyl phenyl ketone Molecular weight, g/mole:204.3 C₁₃H₁₆O₂

Benzophenone Molecular weight, g/mole: 182.2 C₁₃H₁₀O

Irgacure 500 is a mixture of HCPK and BP. At a 1:1 ratio by weight, these two solid photoinitiators form a eutectic mixture that results in a liquid system.

Typical Physical Properties

Appearance Clear, pale yellow liquid

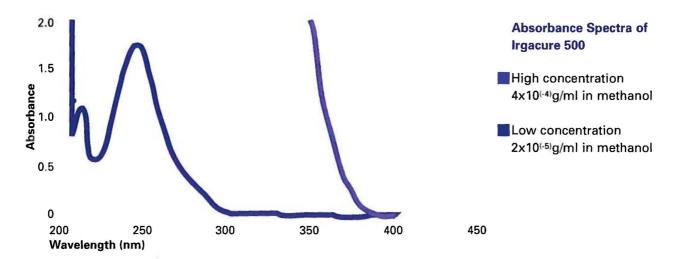
Molecular weight, g/mole 192.62

Melting range, °C Liquid at room temperature

Density, g/ml 1.11

UV absorption peaks, nm 240-260

375-390



Irgacure 651 is a good photoinitiator for general applications where non-yellowing is not important. It can be used for wood, metal and plastic coatings. Irgacure 651 offers good storage stability over benzoin ethers.

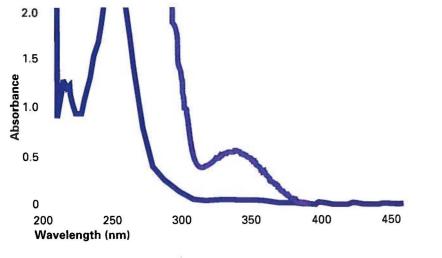
Chemical Structure/Name

2,2-dimethoxy-2-phenyl acetophenone (BDK) $C_{16}H_{16}O_2$

Typical Physical Properties

Appearance White crystalline powder
Molecular weight, g/mole 256.30
Melting range, °C 63-66
UV absorption peaks, nm 330-340

500



Absorbance Spectra of Irgacure 651

- High concentration 5x10⁽⁻⁴⁾g/ml in methanol
- Low concentration 5x10⁽⁻⁵⁾g/ml in methanol

Irgacure 784 DC

Irgacure 784 DC is an efficient visible light photoinitiator. It is a blend of 30% titanocene compound with 70% of an inert clay, Dicalite® 4128. Its absorption spectrum beyond 500 nm makes Irgacure 784 DC a suitable photoinitiator for direct laser imaging applications including photoresists and offset printing plates. The high reactivity of Irgacure 784 DC has been demonstrated by using very low concentrations in different applications.

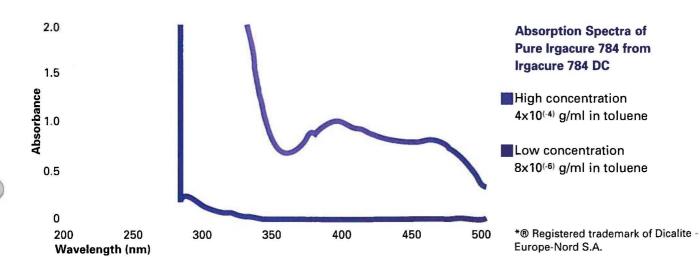
Chemical Structure/Name Bis(η⁵-2,4-cyclopentadien-1-yl)-bis[2,6-difluoro-3-(1H-pyrrol-1-yl)phenyl] titanium

Typical Physical Properties

Appearance
Molecular weight, g/mole
Melting range, °C
UV absorption peaks, nm

Reddish-orange crystal 534.39 190-195 380-390

460-480

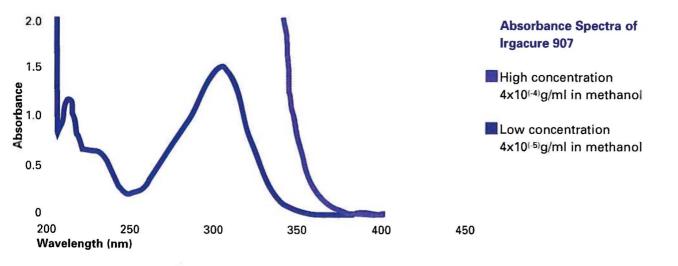


Irgacure 907 is a reactive photoinitiator useful in pigmented systems. Additional sensitizers such as thioxanthones in formulations can enhance the performance of Irgacure 907 significantly. In one study of the curing characteristics of a white coating, it was found that 3% Irgacure 907 gives 10 ft/min curing speed, while an additional 0.3% ITX gives a curing speed at 90 ft/min. It is also known that Irgacure 907 exhibits excellent surface cure due to its high extinction coefficients at the major emission lines of UV curing lamps. The blend of Irgacure 907 and ITX can also be used in white coatings or inks when a non-yellowing requirement is not critical.

Chemical Structure/Name

2-methyl-1-[4-(methylthio)phenyl]-2-morpholino propan-1-one (MMMP) ${\rm C_{15}H_{21}O_2NS}$

Appearance	White to light beige crystalline powder
Molecular weight, g/mole	279.4
Melting range, °C	70-75
Density, g/ml	1.21
UV absorption peaks, nm	320-325



Darocur 1173

Darocur 1173 is a good liquid photoinitiator for use in a wide range of UV curing formulations. It has good solvency properties and can act as a solvent for other photoinitiators. It can also act as a diluent for viscosity reduction. In a viscosity study, Darocur 1173 reduced the viscosity of a clear coating by about 35.5%, while Irgacure 184 gives 27.5% reduction in viscosity.

Chemical Structure/Name

2-hydroxy-2-methyl-1-phenyl-propan-1-one (HMPP) $C_{10}H_{12}O_2$

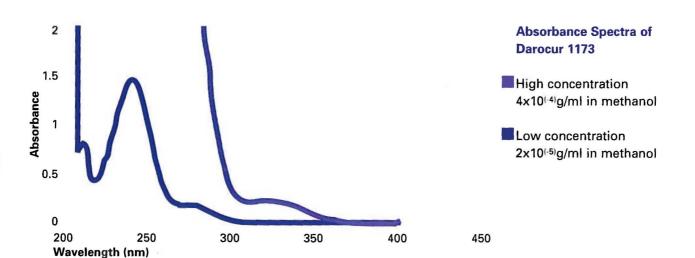
Typical Physical Properties

Appearance Clear, colorless to light yellow liquid

Molecular weight, g/mole 164.2

Boiling point, °C 80-81°C @ 1 mm Hg, 250°C

Density, g/ml 1.074-1.078
UV absorption peaks, nm 265-280
320-335



Irgacure 1700, our newest liquid photoinitiator, is useful in curing pigmented coatings and inks. Its photobleaching effect makes the photoinitiator suitable for white coatings and inks. In addition, Irgacure 1700 has good absorption between 350 nm and 400 nm; therefore, it provides good through cure in highly pigmented white systems.

DMBAPO

25% bis (2,6-dimethoxybenzoyl)-2,4-, 4-trimethylpentyl phosphine oxide Molecular weight, g/mole: 490.0 C₂₆H₃₅O₇P

Typical Physical Properties

Appearance
Molecular weight, g/mole
Melting range, °C
Density, g/ml

UV absorption peaks, nm

HMPP

75% 2-hydroxy-2-methyl-1-phenyl-propan-1-one (HMPP) Molecular weight, g/mole: 164.2 $\rm C_{10}H_{12}O_2$

Clear light yellow liquid

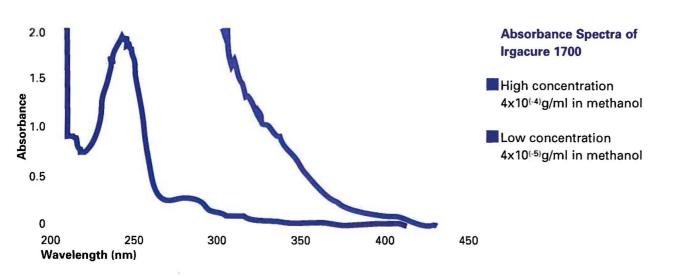
196.96

Liquid at room temp.

1.01

245

325



Irgacure 2959 is a hydroxy ethoxy derivative of Darocur 1173. The hydroxy functional group enhances the compatibility of Irgacure 2959 in UV curable waterborne systems. More importantly, Irgacure 2959 offers the lowest odor in cured films, compared to other commercially available photoinitiators.

$$HO-CH_2-CH_2-O$$
 $\begin{array}{c} O & CH_3 \\ \parallel & \parallel \\ C-C-C-OH \\ \parallel & CH_3 \end{array}$

Chemical Structure/Name

4-(2-hydroxyethoxy)phenyl-(2-hydroxy-2-methylpropyl)ketone $C_{12}H_{16}O_4$

Typical Physical Properties

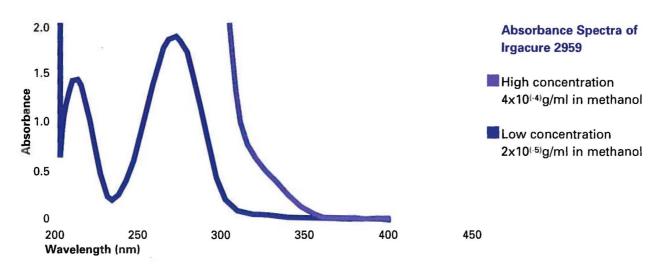
Appearance White crystals

Molecular weight, g/mole 224.26

Melting range, °C 86.5-89.5

UV absorption peaks, nm 275-285

320-330



Darocur 4265

Darocur 4265 is a liquid blend of Darocur 1173 and Lucirin®* TPO, useful in both clear and white pigmented systems. It gives good surface cure when pigment loading or the film thickness is low.

Chemical Composition

Darocur 4265 is a 50:50 blend of 2-hydroxy-2-methyl-1-phenyl-propan-1-one (HMPP) and 2,4,6-trimethyl benzoyl diphenyl phosphine oxide (TPO).

TPO

50% 2,4,6-trimethylbenzoyldiphenylphosphine oxide (TPO) Molecular weight, g/mole 348 C₂₂H₂₁O₂P

Typical Physical Properties

Appearance Molecular weight, g/mole Density, g/ml Melting range, °C

UV absorption peaks, nm

HMPP

 $C_{10}H_{12}O_2$

Clear liquid with slight color 223.20

50% 2-hydroxy-2-methyl-1-

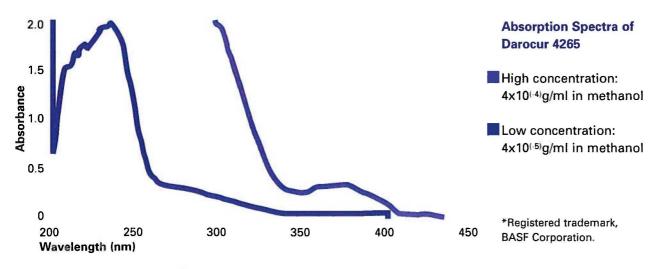
phenyl-propan-1-one (HMPP)

Molecular weight, g/mole 164.2

270-290

Liquid at room temp.

360-380



Other Coatings Additives

UV Absorbers

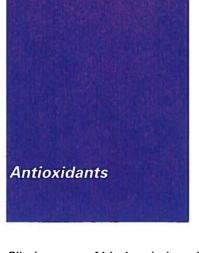
Hindered Amine Light Stabilizers

The appearance and durability of a UV coating may be enhanced through the use of various additives such as UV absorbers, light stabilizers, antioxidants, and optical brighteners. The enhancement due to the additives must be balanced against the potential negative effects they may have on cure rates. For example, a UV absorber may compete with the photoinitiator for the excitation light, decreasing photo speed and affecting other film properties such as hardness.

Ciba pioneered the development of benzotriazole ultraviolet absorbers (UVAs) which are now marketed under the Tinuvin® name. UV absorbers act by absorbing incident UV radiation and converting it to a harmless level of heat energy which is dissipated throughout the coating. A number of products, both liquid and solid, are available from this class and Tinuvin 1130, a liquid UVA, is suggested due to its ease of incorporation.

Because the UV absorber and the photoinitiator compete for the same UV light, significant reductions in cure speed can occur when a UVA is added to the coating. For example, increasing the UV absorber, Tinuvin 1130, concentration from 0% to 2% in a clear coating decreases the photospeed by 30%. For this reason, the UV absorber concentration used should be kept to a minimum or the photoinitiator concentration used should be increased.

A second class of compounds sold under the Tinuvin name, hindered amine light stabilizers (HALS), are efficient stabilizers for coatings. They do not act by absorbing UV light, but rather by scavenging free radicals. HALS may be used alone or in combination with a UVA. HALS are known for their efficiency and longevity as light stabilizers. This is because HALS are regenerated rather than consumed during the stabilization process. Even though HALS do not compete with the photoinitiator for UV light, the additive should be thoroughly tested before inclusion in formulas. Tinuvin 292 in a concentration range of 0.5% to 2.0% is recommended for most applications.



Ciba's group of hindered phenolic antioxidants (AO) are sold under the Irganox® name. Antioxidants hinder thermally-induced oxidation of polymers from which peroxide radicals form. The chemical reactions that occur in high temperature applications can result in the formation of colored chromophores or a change in coating properties. Antioxidants act to terminate peroxide radicals generated during the bake cycle of a coating. Unlike HALS, which regenerate, antioxidants are consumed in the stabilization process. To avoid yellowing, a concentration range of 0.1% to 0.5% is usually recommended.

Optical Brighteners

Optical brightening agents add fluorescence to a coating. This effect can brighten a coating or mask yellowing. These brighteners absorb light in the UV region and emit light in the blue region of the visible spectrum, resulting in a brighter appearance. Since optical brighteners absorb light in the UV region, they may compete with the photoinitiator and reduce cure speeds.

Uvitex® OB, soluble in most UV curable materials, can be used at very low concentrations of between 0.05%-0.5% for a 1 mil coating. It is a highly efficient fluorescent whitening agent which increases coating brightness, masks yellowing or can be used as a tracer. It can also improve the brilliance of inks when used in a clear overprint varnish.

Tinopal® SFP is a water soluble fluorescent whitener. It is used in applications analogous to those of Uvitex OB.

Applications

There are, to date, seven major application areas for UV curing technology. The following section lists these application areas, briefly explaining each.
Recommendations for specific Ciba photoinitiators are given, along with supporting data.



Wood Coatings

Wood coatings, specifically unsaturated polyester wood fillers, represent one of the first commercially successful uses of UV curing technology in the United States. In wood fillers, the photopolymer compounded with silica, talc, and other inerts is applied by a roll coater to fill voids in a particle board substrate. Sandability of the filled substrate then becomes an important physical characteristic of the filler.

Today, use of UV curing technology has expanded far beyond the wood filler application. UV topcoats are now used in prefinished wood flooring, kitchen cabinets, and some three dimensional furniture applications. Growth of UV coatings is due to a number of factors: low VOCs; higher throughput of parts in a given time frame; and excellent abrasion and stain resistance properties. Complete through cure of heavily pigmented wood coatings, e.g. whites, is now possible with the introduction of Ciba's new Irgacure 1700. In addition to excellent through cure properties, this photoinitiator photobleaches after UV exposure to give a clean white. This is in contrast to an off-white color that is associated with other long wavelength absorption photoinitiators.

We recommend:

Application	Film Thickness	Photoinitiator %
Clear Wood Topcoat	0.5 - 4.0 mils	Irgacure 184/1.0-4.0%
		Irgacure 500/2.0-5.0%
		Darocur 1173/1.0-4.0%
		Irgacure 1700/1.0-4.0%
Wood Fillers		
Unsaturated Polyesters	0.5 - 2.0 mils	Irgacure 651/1.0-3.0%
		Irgacure 1700/0.5-2.0%
		Darocur 4265/0.5-2.0%
Acrylates	0.5 - 2.0 mils	Irgacure 184/1.0-3.0%
		Darocur 1173/1.0-3.0%
Pigmented Wood	0.5 - 2.0 mils	Irgacure 1700/1.0-3.0%
Coatings		Darocur 4265/1.0-3.0%

Inks & Pigmented Coatings

UV cured inks are used for a variety of lithographic, flexographic and screen printing applications. These inks present special formulation challenges due to their pigmentation and the need for tightly controlled viscosity.

Many commonly used pigments, such as TiO₂, block part of the UV light spectrum which the photo-initiator needs to cure the ink. This causes a reduction in cure speed and/or prevents a balance between surface and through cure. To solve this problem, Ciba has introduced several photoinitiators which have broader absorption, especially in the longer UV wavelengths.



Irgacure 907 and Irgacure 369 are two such photoinitiators with strong absorption in the longer UV spectrum, and they are very effective in curing blue or black inks. These photoinitiators may be used alone or in combination with a sensitizer such as ITX. Generally, a photoinitiator concentration of 1-4% is suggested with ladder studies to optimize the formula. ITX, when used as a photosensitizer, should be used at 0.25-1% level.

Irgacure 907 and Irgacure 369 may add slight color to the ink. This makes them less preferable for white, pastel and other light colored inks. For this segment of the ink market, a photoinitiator of the phosphine oxide class is suggested. Irgacure 1700 and Darocur 4265 are both blends of a phosphine oxide photoinitiator and Darocur 1173, a liquid photoinitiator of the hydroxyacetophenone class. Both blends have absorption past 400 nm making them

ideal for formulations containing TiO₂ pigment. When reacted, both products photobleach, meaning that the by-products do not absorb in the visible range and thus add color to the ink. Irgacure 1700 is the more reactive of the two products, forming four free radicals instead of two, and making it the more cost effective in many formulas.

Since both Irgacure 1700 and Darocur 4265 are liquids, they can be easily incorporated into the ink at a concentration of 0.5-2%. Additions can be made at almost any point during the formulation process, and post formulation additions to boost cure speed are also possible.

Curing white pigmented coatings is a challenge because of the absorption of rutile TiO₂, a dominant white pigment that absorbs strongly in the UV region up to 380 nm.

In general, combinations of phosphine oxide initiators with other photoinitiators produce harder films than films cured with phosphine oxide alone.

Pigmented Coatings

Early photoinitiators were not very effective in the through cure of pigmented systems. However, developments in the last decade, coupled with continuing research for these systems, have yielded photoinitiators that can successfully be used in the ink and pigmented coatings industries.

Application	Offset Inks	Flexo Inks	Screen Inks	Other
	Film Thickness			
	<0.1 mil	0.1-0.3 mils	0.3-1.0 mils	>1.0 mil
Colors				
White		2-5% I-1700	1-4% I-1700	1-4% 1-1700
	3-6% D-4265	2-6% D-4265	2-4% D-4265	2-4% D-4265
Pastel Colors	3-6% -907*	2-5% I-1700	1-4% I-1700	1-4% I-1700
	3-6% D-4265	2-6% D-4265 2-6% I-907*	2-4% D-4265	2-4% D-4265
Dark Colors	3-6% -907*	2-6% I-907	1-4% I-1700	1-4% l-1700
Dark Colors	2-5% I-369**	1-4% I-369**	1-4% I-369**	1-4% l-369**
	2 0 / 0 . 0 0 0	. 4701 000	2-4% D-4265	2-4% D-4265
Black	3-6% -907*	2-6% I-907*	1-4% l-1700	1-4% I-1700
	2-5% -369**	1-4% I-369**	1-4% I-369**	1-4% I-369**

2-4% D-4265

2-4% D-4265

1-4% I-1700

Ciba Photoinitiator Recommendations for Pigmented Systems

^{*} Can be used with ITX.

^{**} Can be used with Irgacure 184.

Paper Coatings



Paper coatings, particularly overprint varnishes, account for the single largest use of photoinitiators in the United States. Common uses include magazines and consumer goods packaging. UV cured paper coatings are fast becoming the industry choice because, unlike traditionally cured coatings, they exhibit high gloss and excellent abrasion resistance. Paper coatings are run at extremely high speeds (over 200 ft/min per lamp) and they must come off the conveyor belt tack-free to allow for stacking. In general, a dual-component initiator is more efficient in curing a coating to a tack-free state at maximum line speed.

We recommend:

Application

Paper coatings

Film Thickness

.1-.5 mils

Photoinitiator %

Irgacure 500/2.0-8.0% Irgacure 184/1.0-3.0% Darocur 1173/1.0-3.0%

Clear Coatings



Clear coatings encompass a range of applications including metal, wood, glass and plastic. Coating thickness can range from 0.3 mils for cans and up to 4 mils for floor tile and fiber optics. To improve weatherability of UV cured clear coatings, hindered amine light stabilizers (HALS) have proven useful, as they do not interfere with the UV cure process. Gloss retention may also be improved, and

cracking and yellowing minimized by the use of an appropriate HALS in these applications.

We recommend:

Application

Clear coatings on plastic, metal, wood

Film Thickness

1.0-4.0 mils

Photoinitiator %

Irgacure 184/1.0-3.0% Irgacure 500/2.0-4.0% Darocur 1173/1.0-3.0%

Photoresists and Solder Masks

Photoresists are used to define the pattern on a printed circuit board and solder masks are used to ensure that solder is deposited on the board only where required. High cure speed, good performance and the solventless nature of UV cured coatings are factors contributing to the growth of UV cure technology in the electronics industry.

We recommend:

Application

Photoresist/solder

masks

Film Thickness Photoinitiator %

0.2-2.0 mils Irgacure 907/1.0-5.0% Irgacure 369/1.0-5.0%

Printing Plates

Photopolymer printing plates, the dominant technology for printing newspapers, are also being used in flexographic printing. These plates range in thickness from 15-250 mils. For this application, photoinitiators with exceptional curedepth are required.

We recommend:

Application

Printing plates

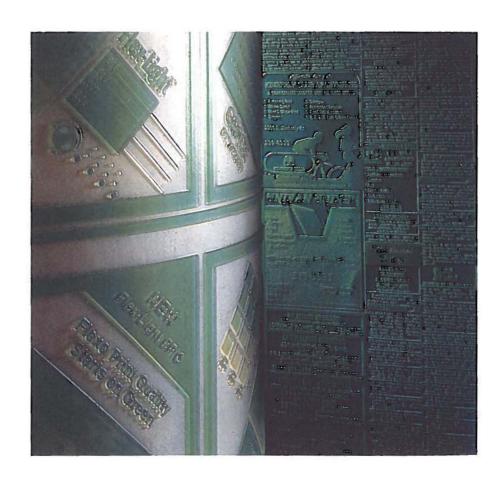
Film Thickness

15-250 mils

Photoinitiator %

Irgacure 651/1.0-3.0%

Irgacure 369/.5-2.0%



Adhesives

UV-cured adhesives is a broad category encompassing laminates for wood, paper, plastics, glass and metal. In addition, there are specialty applications for electronics, medical devices and automotive uses. In most of these applications, the adhesive film is relatively thick and requires longer exposure times in order to achieve acceptable through cure. For this reason, a photoinitiator with a relatively low extinction coefficient at the wavelength of the light source is required.

Several Ciba photoinitiators have the ability to cure thicker sections. Irgacure 369, Irgacure 907, and Irgacure 1700 are the photoinitiators of choice since they have absorption curves that extend out past the long UV and into the short visible region. Matching of the light source to the photoinitiator absorption is critical here. A bulb is needed that emits in the range of 350 nm to 430 nm to help optimize the curing process. This can be especially critical if the exposure is made through a film which absorbs the short UV radiation.

Photoinitiator concentrations of 1-4% are suggested and blends with shorter wavelength absorbing photoinitiators such as lrgacure 184 or Darocur 1173 may be needed to get a balance of through and surface cure.

We recommend:

Application	Film Thickness	Photoinitiator %
Pressure-sensitive adhesives	0.5-5.0 mils	Irgacure 184/1.0-4.0%
Laminating adhesives	0.3-2.0 mils	Irgacure 1700/.5-3.0%
Structural adhesives	Variable	Irgacure 369/1.0-4.0%
		Darocur 1173/1.0-4.0%

Thick Section Curing

Thick section curing covers a wide area: sealants, adhesives, printing plates and potting compounds. The polymer in most of these applications must have a relatively long dwell time under the UV light source and be stationary or moving at very low line speed. Good through cure here requires efficient penetration of light down to the polymer substrate interface. Therefore, the chosen photoinitiator must have a relatively low extinction coefficient at the wavelengths of the light source.

We recommend:

Application

Variable

Film Thickness

>5 mil

Photoinitiator %

Irgacure 369/.5-2.0% Irgacure 1700/.5-2.0% Darocur 4265/.5-2.0% Irgacure 184/1.0-3.0%

Physical Properties of Ciba Photoinitiators

Ciba Photoinitiator Weight Loss/Volatility Characteristics

Table 2
Weight Loss/Temperature (°C) Profile of Photoinitiators by TGA*

	% Weight Loss		
Photoinitiator	5%	10%	15%
Benzophenone	153°	167°	176°
Irgacure 184	155	170	179
Irgacure 261	227	246	256
Irgacure 369	248	264	274
Irgacure 651	170	184	194
Irgacure 784	213	217	220
Irgacure 907	198	214	224
Irgacure 2959	216	233	243

^{*} Heating rate 10°C/min.

Table 3Ciba Photoinitiator Volatility Study
ASTM D-2369 Test Method

		% Weight Loss
Photoinitiator	Neat	10 wt% in TMPEOTA
Darocur 1173	98.6	8.6
Benzophenone	26.6	4.3
Irgacure 500	25.9	2.8
Irgacure 184	17.4	2.6
Irgacure 651	7.0	2.8
Irgacure 2959	8.0	0.0
Irgacure 907	0.7	0.0
Irgacure 261	0.1	0.0
Irgacure 369	0.0	0.0
Conditions:	0.5 g. sample in 2 ml Toluene	
	60 minutes @ 110 +/- 5 °C	

Photoinitiator Solubility/Chemical Class

Table 4
Solubility (wt %) of Ciba Photoinitiators

	Irg. 184	lrg. 369	lrg. 651	lrg. 907	lrg. 2959
IBOA	≥50%	15%	40%	35%	5%
IDA	≥50%	5%	30%	25%	5%
PEA	≥50%	15%	≥50%	45%	5%
HDDA	≥50%	10%	40%	35%	10%
TRPGDA	≥50%	10%	25%	30%	20%
TMPTA	≥50%	5%	≥50%	25%	5%
TMPEOTA	≥50%	5%	45%	20%	5%
CHVE	≥50%	10%	≥50%	40%	≥50%
DVE-3	≥50%	10%	45%	40%	≥50%
Darocur 1173	≥50%	30%	≥50%	≥50%	35%

Note: The solid photoinitiators were dissolved in liquid monomers (by weight ratio), heated with a warm water bath (50-60°C) and mixed. Samples were then kept at room temperature for 24 hours. If no recrystallization occurred, results were then recorded. Only 5% increments of photoinitiators were employed in the study. For example, the solubility of Irgacure 907 in IBOA is 35% which means that its solubility is above 35% but below 40%.

Table 5 Ciba Photoinitiators by Class

alpha-Hydroxy- ketones	alpha-Amino- ketones	Benzildi- methyl ketal	alpha-Hydroxy- ketone blends	Metallocene
Irgacure 184	Irgacure 907	Irgacure 651	Irgacure 500	Irgacure 261
Darocur 1173	Irgacure 369		Darocur 4265	Irgacure 784
Irgacure 2959			Irgacure 1700	

Important

These materials are not intended for use in products for which prolonged contact with mucous membranes or abraded skin, or implantation within the human body, is specifically intended, unless the finished products have been tested in accordance with the Food and Drug Administration and/or other applicable safety testing requirements. Because of the wide range of such potential uses, Ciba is not able to recommend this material (these materials) as safe and effective for such uses and assumes no liability for any such use.

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