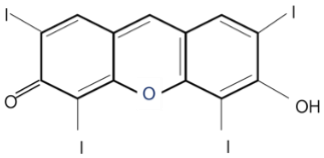
Technical Data Sheet

H-Nu 535

Visible/UV-Visible
Light Photoinitiator

Product Description

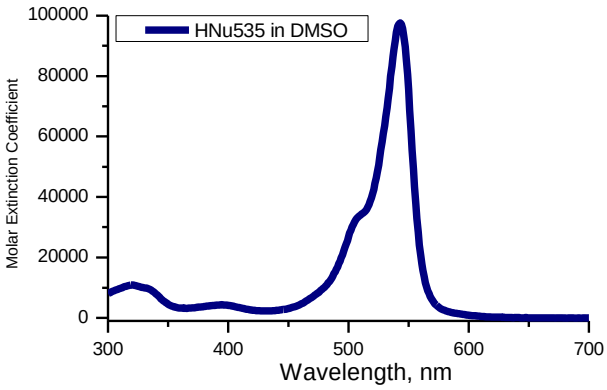


Name: 2,4,5,7-tetraiodo-3-hydroxy-6-fluorone
CAS # 142189-38-6
Formula: $C_{13}H_3I_4O_3$
Synonyms: TIHF, H-Nu 535

- Initiates both free-radical and cationic photopolymerization, using a different set of co-initiators depending on the polymerization process
- Type II photoinitiator: H-Nu 535 acts as a light harvester, followed by energy or electron transfer to a co-initiator molecule; produced reactive species activate photopolymerization
- Commercial photoinitiator, non-toxic (LD50>2500 mg/kg)

Advantages

- ∨ High absorptivity, low concentrations are needed (0.01-0.15% by weight)
- ∨ Cationic cured epoxides are not susceptible to oxygen inhibition and exhibit a tack free surface upon cure
- ∨ Capable of significant depth of cure > 1 inch
- ∨ Cure through UV opaque or pigmented substrates (e.g. Kapton)
- ∨ Initiator bleaching (bright red to pale pink/no color) upon light interaction can act as cure indicator



Typical Properties

Property	Test Method	Typical Value
Absorbance Maximum	UV Visible spectroscopy	535 nm
Appearance	Visual	Red Powder
Melting point	Melting point Analysis	>270 °C
Molar Extinction Coefficient	UV Visible spectroscopy	91,200 (at 535nm)

Applications Recommendations

∨ Direct solubility of H-Nu 535 in resins can be difficult, pre-dissolution in one of the following resins/solvents before adding resin is recommended:

Free-Radical Diluents						Cationic Diluents				
DMAA	VMOX	THFA	HDDA	HPMA	IBOA	Heloxy61	Heloxy62	EOXA[1]	ECC[2]	Eponex1510
S	S, T°	I	I	I	I	I	I	I	P, T°	S, T°

1% of H-Nu 535 in diluent is: S-soluble T°-requires heat (60°C for several hours) P-partially soluble I-insoluble
1 = 3-ethyl-3-hydroxymethyloxetane
2 = 3,4-epoxycyclohexylmethyl-3,4

Note: DMAA is an excellent solvent for H-Nu 535, but it **cannot** be used in **cationic polymerization** as it inhibits process.

∨ The presence of any undissolved red particles is an indication of incomplete solubility. More heating (60°C) or stirring may help with incorporation, or pre-dissolution using different diluents is recommended.

Polymerization mechanisms

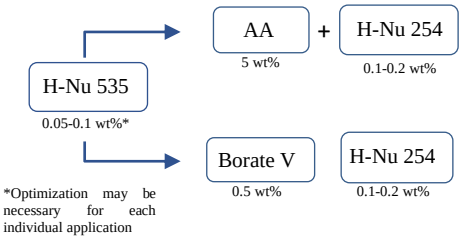
Free-Radical Cure (Acrylates)

∨ H-Nu 535 concentration range spans from 0.05 to 0.2 wt.%, depending on a cure thickness. 0.05 wt% is recommended starting point for thick cure (>1mm) and 0.1wt% for thin cure.

∨ Amine acrylates (AA) are needed as co-initiators at 5-10 wt.%. If amine use is counterindicated (acidic adhesion promoter, outgassing), as an alternative, another co-initiator, Borate V (0.5 wt.%) may provide similar cure response as typical amine co-initiators

∨ H-Nu 254 iodonium salt is recommended for acceleration.

Recommended starting concentrations:



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Cationic Cure (Epoxides)

- H-Nu 535 concentration range spans from 0.05 to 0.2 wt.%, with a good starting point at 0.1 wt.% based on solids.
- H-Nu 254 iodonium salt is needed to achieve cure in epoxide resins (sulfonium salts will not work).
- Accelerator AN-9,10-E can greatly enhance cure speed and sensitivity and is needed to achieve cure.
- Amines or DMAA solvent cannot be used due to the superacid "poisoning" effect.
- Caution & experimentation is required at higher temperatures as it can lead to thermal ground state polymerization. Additional stabilizers, depending on the photopolymerization matrix can be used. As an example, 0.2 wt.% of PVP+BHT* mixture (1:1) can be added to glycidyl epoxide and 0.3 wt.% of PVP+BHT mixture (1:1) to cycloaliphatic epoxide.

Recommended starting concentrations:

H-Nu535	+	H-Nu254	+	AN-9,10-E	+	BHT/PVP
0.05-0.1 wt%*		2.5 wt%		0.05-1 wt%		0.2-0.3 wt%

*Optimization may be necessary for each individual application
PVP = Polyvinylpyrrolidone
BHT = Butylated hydroxytoluene

Examples of H-Nu 535 uses:

SU-8 photoresist

- Y. Lin, P.R. Hermann, and K.Darmawikarta, Appl. Phys. Lett. 86, 7, 071117 (2005)
- J.H. Moon, S.-M. Yang, D.J. Pine, and W.-S. Chang, Appl. Phys. Lett. 85, 18, 4184 (2004)
- D. Rodriguez Ponce, K Lozano, et al. J. Polym. Sci.: Part B: Polym. Phys. 48, 1, 47 (2010)

3D printing

- Neil D. Dolinski, Zachariah A. Page, et al. J. Adv. Mater. 30, 31, 1800364 (2018)
- Neil D. Dolinski, E. Benjamin Callaway, et. al. ACS Appl. Mater. Interfaces 13, 22065-22072 (2021)
- Luke F. Gockowsi, Neil D. Dolinski, et. al. Mater. Horiz., (2020), DOI: 10.1039/D0MH00331J

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