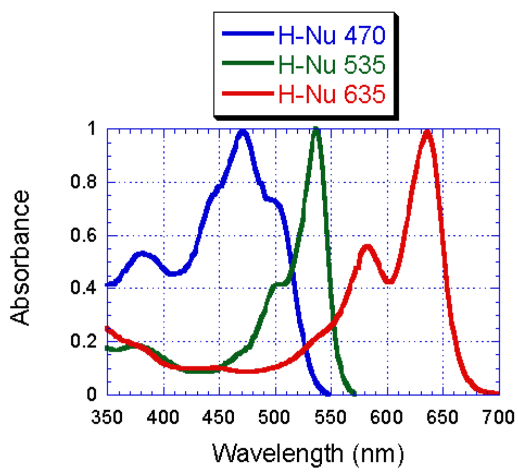
H-Nu 470 Visible/UV-Visible Light Photoinitiator and H-Nu 535 Visible Light Photoinitiator for Cationic Epoxide Curing

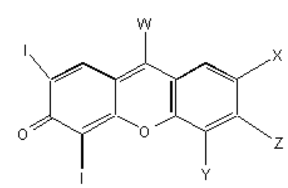
Synonyms: H-Nu 470, DIBF	**Formula: $C_{17}H_{14}I_2O_3$**
Name: 5,7-diiodo-3-butoxy-6-fluorone	**CAS # 161728-47-8**
Synonyms: H-Nu 535, TIHF	**Formula: $C_{13}H_3I_4O_3$**
Name: 2,4,5,7-Tetraiodo-3-hydroxy-6-fluorone	**CAS # 142189-38-6**

General Information

- **H-Nu Series of Fluorone dyes** (**H-Nu 470, 535**) - photoinitiators with panchromatic absorbance throughout the UVA/visible spectrum (350-550 nm)
- **H-Nu 470 and 535** - capable of curing Epoxide resins via a cationic mechanism
- **H-Nu 470** - commercial photoinitiator, broad absorbance range of 350 nm to 530 nm (λmax=470 nm); (LVE from the EPA), non-toxic (LD50>5000mg/kilo)
- **H-Nu 535** - experimental photoinitiator, broad absorbance range of 450 nm to 550 nm (λmax=535 nm); LD50 > 2500mg/kg (mouse)



H-Nu Photoinitiator Structures



Compound	W	X	Y	Z	ε	λmax (nm)
H-Nu 470	H	H	H	OBu	30200	470
H-Nu 535	H	I	I	OH	91200	535
H-Nu 635	CN	I	I	OH	80000	635

ε is the molar extinction coefficient   λmax (nm) is the peak maximum absorbance wavelength

Benefits of Use

- High absorptivity, low concentrations are needed (0.01-0.15 wt%)
- Cationic cured epoxides are not susceptible to oxygen inhibition and exhibit a tack free surface upon cure
- Cure through UV opaque, pigmented, or colored substrates (e.g. Kapton)
- Accelerated cure with the addition of **AN-910-E** coinitiator

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Physical Properties

Photoinitiator	H-Nu 470	H-Nu 535
Appearance	Orange Powder	Red Solid
Molecular Weight	520 g/mol	716 g/mol
Melting Point	>270 °C	>270 °C
Absorbance Maximum	470 nm	535 nm
Molar Extinction Coefficient	30,200 (470 nm)	91,200 (535 nm)

Photoinitiator Usage Recommendations

Complete dissolution of H-Nu Photoinitiators is required for best results:

- Dissolving H-Nu photoinitiators requires special care. Direct solubility of H-Nu 470/535 in resins can be difficult so predissolution of H-Nu photoinitiators in γ–**Butyrolactone** before adding resin is recommended
    - **GBL (**γ–**Butyrolactone**) – use at 5-10 parts **GBL** to 1 part **H-Nu 470/535**
- If not predissolving **H-Nu 470/535**, <u>stirring/heating (65C is ok) at least 3 to 4 hours</u> before using the formulation to ensure maximum solubility is recommended. The presence of any undissolved orange particles is an indication of incomplete solubility.
- **H-Nu photoinitiator** systems and materials that contain them are <u>light sensitive</u> and should be <u>kept in the dark or in light proof bottles when not in use</u>.

Photopolymerization Mechanisms

Cationic Cure – Epoxides (including SU-8 photoresists*)

- **H-Nu 470/535** requires **H-Nu 254 Iodonium salt** to achieve cure in epoxide resins
- **Accelerator AN-910-E** can <u>greatly enhance cure speed and sensitivity</u> and is recommended, but not required to achieve cure
- **H-Nu 470/535** concentration range spans from 0.05 to 0.2 wt.%, with a good starting point at 0.10 wt.% based on solids
- Recommended starting concentrations:
  Standard: 0.1 wt.% H-Nu 470/535 + 2.5 wt.% H-Nu 254 Iodonium Salt

  With Accelerator: 0.1-0.2 wt.% H-Nu 470/535 + 0.1-1 wt.% AN-910-E + 2.5-3 wt.% H-Nu 254 Iodonium Salt
- Sulfonium salts <u>will not work</u> with **H-Nu 470/535** – use an iodonium salt
- Do **<u>NOT</u>** use **Amines** or **DMAA (N,N-Dimethylacrylamide)** solvent - amines "poison"/quench the superacid formation, preventing cationic cure

***SU-8 w/470 References:** 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)

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Formulation Examples

Cationic Polymerization

Structural adhesive

0.1%   H-Nu 470/535
0.1%   AN-910-E
2.5%   H-Nu 254 Iodonium Salt
73%   EPON 828 (bisphenol A diepoxide)
24.3%   Tone 305 (polyol)

Two corona treated polyethylene films - cure time 30-40 seconds, room temperature - Xenon RC-500 Lamp OR a 360W Tungsten halogen lamp – 1" distance. Cohesive failure of polyethylene substrate before the adhesive bond fails.

Flexible composite

0.1%   H-Nu 470/535
0.1%   AN-910-E
2.5%   H-Nu 254 Iodonium Salt
97.3%   Proprietary epoxide resin

Free standing film – cure time 30-60 sec, room temperature - 360 W tungsten halogen visible light - 1" distance.

SU-8 Photoresist

0.1%   H-Nu 470/535 (based on SU-8 resist solids)
2.5%   H-Nu 254 Iodonium Salt (based on SU-8 resist solids)
1wt%   AN-910-E (optional, based on SU-8 resist solids)
X%   SU-8 resist solids
Y% γ-butyrolactone solvent (X and Y% are determined by the user based on the desired %solids for the spincoating film deposition operation)

SU-8 Material widely used in the electronics industry; curable using UV or visible light via cationic mechanism. SU-8 is a solid that must be dissolved in a solvent such as γ-butyrolactone (γ-BL). H-Nu photoinitiators, H-Nu 254 iodonium salt and AN accelerator are quite soluble in γ-BL which allows for good incorporation into the formulation. Sulfonium salt needed for UV in SU-8 may remain for visible light process. AN-910-E cationic accelerator leads to faster imaging and stronger photoresist structures formed. For best results the ladder of H-Nu 470 or H-Nu 535/H-Nu 254 iodonium salt /AN-910-E has to be run.

After spincoating prebake is required to remove all solvent from the film. **DO NOT** exceed 110C on prebake.

After imaging brief postbake may be required to maximize epoxide conversion in imaged areas for sharper contrast.

Product Safety and Handling

Please read MSDS information before handling any products described in this brochure.

Disclaimer

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