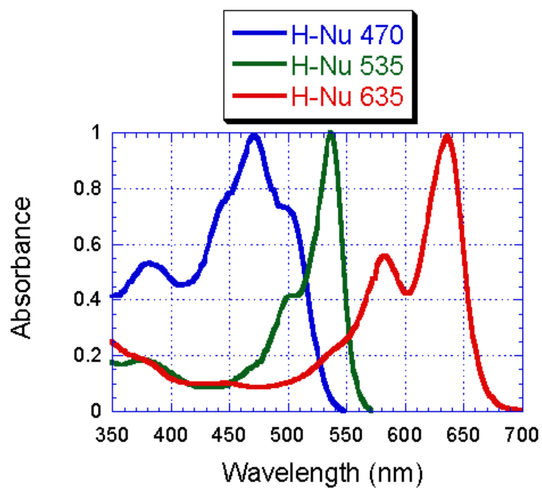
H-Nu 535 Visible Light Photoinitiator

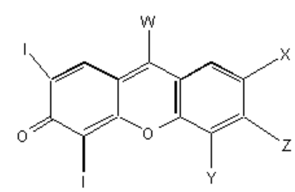
Name: 2,4,5,7-Tetraiodo-3-hydroxy-6-fluorone	Formula: $C_{13}H_3I_4O_3$
CAS # 142189-38-6	Synonyms: TIHF, H-Nu 535

General Information

- H-Nu Series of Fluorone dyes (**H-Nu 470, 535 and 635**) - photoinitiators with panchromatic absorbance throughout the UVA/visible spectrum (350-670 nm)
- **H-Nu 470, 535 and 635** - capable of curing a wide range of resins:
  - Acrylates - free-radical mechanism
  - Epoxides - cationic mechanism
- **H-Nu 535** - experimental photoinitiator, broad absorbance range of 450 nm to 550 nm (λmax=535 nm)
- Non-toxic -- 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%)
- Capable of significant depth of cure in free radical formulations, > 1 inch
- Time and energy savings when one-pass thick cure can replace thin multi-layered coatings
- Cure through UV opaque, pigmented, or colored substrates (e.g. Kapton)
- Initiator bleaching: from bright red to pale orange/no residual color
- Bleaching/color change indicator of exposure/cure with UV/visible light

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Physical Properties

Appearance	Red Solid
Molecular Weight	716 g/mol
Melting Point	>270 °C
Absorbance Maximum	535 nm
Molar Extinction Coefficient	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 535 in resins can be difficult, predissolution of H-Nu photoinitiators in one of the following resins/solvents before adding resin is recommended
    - **Free Radical Only** -- **DMAA (N,N-Dimethylacrylamide)**
        - usage at 5-10 parts **DMAA** to 1 part **H-Nu 535**
    - **Cationic Only** – **GBL (γ–Butyrolactone**)
        - Usage at 5-10 parts **GBL** to 1 part **H-Nu 535**

  NOTE: **DMAA** is an excellent solvent for **H-Nu 535**, but it **cannot be used in cationic resins** as it inhibits cure.

- The presence of any undissolved red particles is an indication of incomplete solubility. More heating or stirring may help with incorporation, or predissolution using **DMAA** or **GBL** as noted above is needed.

- Predissolving **H-Nu 535** in the appropriate material may allow for easier addition and faster usage. If not predissolving **H-Nu 535**, stirring/heating (65C is ok) at least 3 to 4 hours before using the formulation to ensure maximum solubility is recommended.

- **H-Nu photoinitiator** systems and materials that contain them are light sensitive and should be kept in the dark or in light proof bottles when not in use.

- "Dimmed" light conditions or other form of light shielding for mixing and formulating when using H-Nu photointiators are recommended to prevent unwanted pre-polymerization.

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Photopolymerization Mechanisms

Acrylate Cure (Free-radical)

- Coinitiators are required – amine acrylates (**AA**) at 5 - 10 wt.% are recommended
- H-Nu 254 iodonium salt is recommended for acceleration if needed (may cause instability)
- Recommended starting level of **H-Nu 535** - 0.05 - 0.15 wt.% based on total solids.
- Recommended starting concentrations:

Thin Cure (< 1 mm)

0.10 wt.% **H-Nu 535**	0.15 wt.% **H-Nu 254**	5 wt.% **Amine Acrylate (AA)**

Thick Cure (1 mm or greater)

0.05 wt.% **H-Nu 535**	0.15 wt.% **H-Nu 254**	5-10 wt% **AA**
0.05 wt.% **H-Nu 535**	N/A	5-10 wt.% **AA**

Optimization may be necessary for each individual application

SGL's experimental coinitiator **Borate V** improves cure response over typical amine coinitiators and can be purchased separately.

Photoinitiator package (when added to a model acrylate formulation)	**Reactivity (1-highest, 5-lowest)**	**Stability and storage**
H-Nu 535 + H-Nu 254 + Borate V	**1**	Needs refrigeration as it may polymerize in the dark at room T, can be used by mixing just prior to using.
H-Nu 535 + Sulfonium Hexafluroantimonate Salt + Borate V	**2**	Needs refrigeration as it may polymerize in the dark at room T, can use by mixing just prior to using.
H-Nu 535 + Borate V	**3**	Stable at room T, refrigeration recommended when not in use to prolong shelf life.
H-Nu 535 + H-Nu 254 + Amine	**4**	May be unstable depending on resin used – use only as necessary
H-Nu 535 + Amine	**5**	Stable

- Typical formulations with Borate V:

0.05 - 0.1 wt.% **H-Nu 535**	N/A	0.50 wt.% **Borate V**
0.05 – 0.1 wt.% **H-Nu 535**	0.5 wt.% **H-Nu 254**	0.50 wt.% **Borate V**
0.05 – 0.1 wt.% **H-Nu 535**	1 wt.% **Sulfonium Salt**	0.50 wt.% **Borate V**

It is best to dissolve **Borate V** directly into **DMAA** (2 parts **DMAA** to 1 part **Borate V**) before adding the resin as **Borate V** is difficult to dissolve in some resin systems.

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

- **H-Nu 535** requires **H-Nu 254 Iodonium salt** to achieve cure in epoxide resins
- **Accelerator AN-910-E** can greatly enhance cure speed and sensitivity and is recommended
- **H-Nu 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:
  0.10 wt.% H-Nu 535 + 2.5 wt.% H-Nu 254 Iodonium Salt

  With Accelerator:
  0.10 wt.% H-Nu 535 + 0.1 wt.% AN-910-E + 2.5 wt.% H-Nu 254
- Sulfonium salts will not work with **H-Nu 535** – you must use an iodonium salt
- Do not use **Amines** or **DMAA** solvent as they "poison" or quench the superacid formation, thus preventing cationic cure

***SU-8 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)

Formulation Examples

Free Radical Polymerization: All values are wt%

Thin film with LCD projector activation:

0.3% H-Nu 535
2% DMAA
2% DIDMA (free amine)
0.66% H-Nu 254
Photoinitiator package added to:
5% Fumed silica, thixotrope
95% Acrylate resin mix (consists of 15 parts CN964E75, 40 parts SR 454, 20 parts SR 306, 15 parts SR 399)

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Cationic Polymerization

Structural adhesive

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

Flexible adhesive

0.1%	H-Nu 535
0.1%	AN-910-E
2.5%	H-Nu 254
97.3%	Proprietary epoxide resin

Product Safety and Handling

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

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