

H-Nu 470 Visible/UV-Visible Light Photoinitiator

| Name: 5,7-diiodo-3-butoxy-6-fluorone | Formula: | C ₁₇ H ₁₄ I ₂ O ₃ |
|--------------------------------------|-----------|---|
| CAS # 161728-47-8 | Synonyms: | DIBF, H-Nu 470 |

General Information

- <u>H-Nu Series of Fluorone dyes</u> (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 470 commercial photoinitiator, broad absorbance range of 350 nm to 530 nm (λmax=470 nm)
- Commercially available (LVE from the EPA), non-toxic (LD50>5000mg/kilo)
- H-Nu 470 and its use is protected by U.S. Patents 5,451,343 and 5,395,862



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 multilayered coatings
- Cure through UV opaque, pigmented, or colored substrates (e.g. Kapton)
- Initiator bleaching: from bright orange to pale yellow/no residual color
- Bleaching/color change indicator of exposure/cure with UV/visible light

27800 Lemoyne Road Suite J Millbury, OH 43447, USA Ph: 419-837-9783 Fax: 419-837-6816

Email: info@sglinc.com Web: www.sglinc.com

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Physical Properties

AppearanceOrange PowderMolecular Weight520 g/molMelting Point>270 °CAbsorbance Maximum470 nmMolar Extinction Coefficient 30,200 (470 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 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 470
 - Cationic Only GBL (γ Butyrolactone)
 - Usage at 5-10 parts GBL to 1 part H-Nu 470

NOTE: DMAA is an excellent solvent for **H-Nu 470**, but it **cannot be used in cationic resins** as it inhibits cure.

- The presence of any undissolved orange 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.
- <u>Predissolving</u> H-Nu 470 in the appropriate material <u>may allow for easier</u> addition and faster usage. If not predissolving H-Nu 470, <u>stirring/heating</u> (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 <u>light</u> sensitive and should be <u>kept in the dark or in light proof bottles when not</u> 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)
- When used in combination with common UV initiators and a typical Hg Arc • curing device, cured coating properties are enhanced allowing the user to reduce the amount of UV initiator. Better cure depth is achieved:
 - 1) Moore, M., Lungu, V., Marino, T., Radtech Report 11, 2, (March/April) 1997; 2) US Patent 6,211,262 "Corrosion Resistant, Radiation Curable Coating",
 - Mejiritski, A., Marino, T, Lungu, V., Martin, D., Neckers, D. C.)
- Recommended starting level of **H-Nu 470** 0.05 0.15 wt.% based on total solids. •
- Recommended starting concentrations: •

Thin Cure (< 1 mm)

| 0.10 wt.% H-Nu 470 | 0.15 wt.% H-Nu 254 | 5 wt.% Amine Acrylate |
|--------------------|--------------------|-----------------------|
| | iodonium salt | (AA) |

Thick Cure (1 mm or greater)

| 0.05 wt.% H-Nu 470 | 0.15 wt.% H-NU 254 iodonium salt | 5-10 wt% AA | | |
|---|-------------------------------------|--------------|--|--|
| 0.05 wt.% H-Nu 470 | N/A | 5-10 wt.% AA | | |
| Optimization may be necessary for each individual application | | | | |

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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 470 + H-NU 254 iodonium salt + 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 470 + 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 470 + Borate V | 3 | Stable at room T, refrigeration recommended when not in use to prolong shelf life. |
| H-Nu 470 + H-NU 254 iodonium salt | 4 | May be unstable depending on resin |
| + Amine | | used – use only as necessary |
| H-Nu 470 + Amine | 5 | Stable |

Typical formulations with **Borate V**:

| 0.05 - 0.1 wt.% H-Nu 470 | N/A | 0.50 wt.% Borate V |
|--------------------------|-----------------------|--------------------|
| 0.05 – 0.1 wt.% H-Nu 470 | 0.5 wt.% H-NU 254 | 0.50 wt.% Borate V |
| | iodonium salt | |
| 0.05 – 0.1 wt.% H-Nu 470 | 1 wt.% Sulfonium Salt | 0.50 wt.% Borate V |

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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.

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

- H-Nu 470 requires H-Nu 254 lodonium salt to achieve cure in epoxide resins
- Accelerator AN-910-E can greatly enhance cure speed and sensitivity and is recommended
- H-Nu 470 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 470 + 2.5 wt.% H-Nu 254 lodonium Salt

With Accelerator: 0.10 wt.% H-Nu 470 + 0.1 wt.% AN-910-E + 2.5 wt.% H-Nu 254 lodonium Salt

- Sulfonium salts <u>will not work</u> with H-Nu 470 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 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)

Product Safety and Handling

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

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