Photochemistry in Flow

Vapourtec Ltd

The UV-150 Advanced Photochemical Reactor
March 2014 Vapourtec launched the UV-150 continuous flow photochemical reactor

"Bringing photochemistry to the bench"
“What makes it special and how does it work?”

- Temperature control -40°C to 80°C
- Wavelength selection 250 nm to 700 nm
- Precise control of exposure time
- Safe, quick & easy to set up and use
- Efficient reactor design throughput up to 15 grams / hour

At the heart of the UV-150 is the compact reactor housing

“What’s inside…….”
Light sources available

Vapourtec: UV-150 light sources

Low pressure mercury – 3 options:
- 254 nm
- 310 nm
- 370 nm

Medium pressure mercury:
220 nm to 600 nm
Filters to select desired wavelength

LEDs a range of precise wavelengths:
365 nm to 700 nm
Reactor construction from centre out

Medium pressure mercury lamp

Filter used to select desired wavelength range (9 options)
Lamp and filter zone

Quartz “barrier” tube

Ambient air used to cool lamp and filter by forced convection

Warm air vented safely after exiting lamp / filter zone
Temperature control of the reactor zone

Compact reactor. 1.3mm bore fluoropolymer tube. Internal volumes 2 ml to 10 ml.
Temperature control of the reactor zone

Gas from the reactor zone is exhausted separately to the air from the lamp zone.

Quartz “outer” tube separates reactor from heat sink.

High flow rate of gas at controlled temperature controls reactor temperature.

Temperature sensor in contact with the reactor’s surface.
Heat management in the UV-150

Dichroic mirror allows >450nm to pass through while reflecting UV wavelengths.

Heat passing through the dichroic mirror is conducted away through the reactor body.

Optional fibre optic spectrometer to monitor absorption of photons during the reaction.
Transmission of the fluoropolymer reactor
Vapourtec: Light sources

Low pressure mercury lamp (3 options)
Vapourtec: Light sources

LED lamps (15 options)
Matching LED wavelength to the catalyst

Tetraphenylporphyrin, [TPP] in toluene

Molar Extinction (cm\(^{-1}\)/M)

Wavelength (nm)

420 nm LED
Vapourtec: Light sources

Pure mercury medium pressure lamp (unfiltered)
Comparison of long-pass filters (5 options)

- Filter #1 (quartz)
- Filter #3 (pyrex)
- Filter #7
- Filter #8
- Filter #9

Normalized transmission vs. Wavelength (nm)
Comparison of band-pass filters (4 options)
The UV-150 has been cited in diverse publications

Reevaluation of the 2-nitrobenzyl protecting group for nitrogen containing compounds: an application of flow photochemistry

Photodecarboxylative Benzylations of N-Methoxyphthalimide under Batch and Continuous-Flow Conditions

Light-Induced C - H Arylation of (Hetero)arenes by In Situ Generated Diazo Anhydrides

Batch and Flow Synthesis of Pyrrolo[1,2-a]-quinolines via an Allene-Based Reaction Cascade
From polymer synthesis to protection group removal

Photodecarboxylations in an advanced meso-scale continuous flow photoreactor

Continuous flow photo-initiated RAFT polymerisation using a tubular photochemical reactor

Automated glycan assembly of xyloglucan oligosaccharides

Continuous photochemistry: the flow synthesis of ibuprofen via a photo-Favorskii rearrangement
Baumann, M., & Baxendale, I. R. (2016). Reaction Chemistry & Engineering
Researchers based geographically from Canada to China

Visible-Light Photoredox Catalysis using a Macromolecular Ruthenium Complex: Reactivity and Recovery by Size-Exclusion Nanofiltration in Continuous Flow

Continuous flow photochemistry as an enabling synthetic technology: synthesis of substituted-6(5H)phenanthridinones for use as poly (ADP-ribose) polymerase inhibitors

Photoactive and metal-free polyamide-based polymers for water and wastewater treatment under visible light irradiation

Efficient metal-free photochemical borylation of aryl halides under batch and continuous-flow conditions.
Illuminating immobilised photo catalysts

Another new photochemical reactor under development by Vapourtec Ltd.

Vapourtec: Enabling Photochemistry

- Illumination by LED applied around column
- Temperature control -20°C to 80°C
- Wavelength range 365nm to 700 nm