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Continuous-flow photochemistry made easy with Vapourtec's photoreactor series.

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Recently, continuous-flow operation has emerged as a new technology trend in synthetic organic photochemistry, especially on laboratory scales [1]. Compared to traditional batch systems such as immersion-well or chamber reactors, flow photoreactors offer a range of advantages:

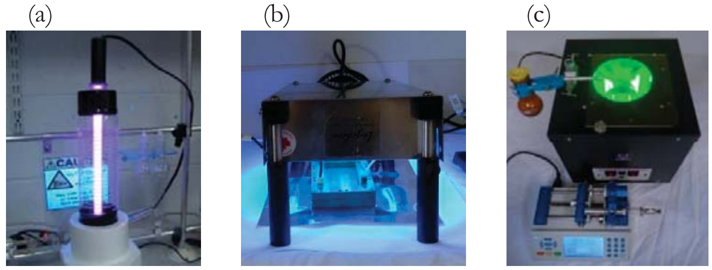
- effective light penetration through the reaction medium,
- removal of (light-sensitive) products from the irradiated zone,
- greater heat and mass transfer within the small reactor dimensions,
- safer operations under higher temperatures and pressures, and
- easy reaction scalability from process optimization to demonstration-scale production.

These representative benefits make continuous flow an attractive option with the potential to implement preparative photochemistry for pharmaceutical applications and fine chemical productions.

Many flow photoreactors are largely improvised devices with components taken from conventional flow and photochemical technologies (**Figures 1**). Commercial flow devices in combination with self-fabricated light-sources are also common. These systems generally suffer from compromised designs, incomplete descriptions and inadequate characterizations. This has led to a generally poor reproducibility of the reactors themselves as well as the experimental results achieved within these devices [2].

With the growing importance and acceptance of flow photochemistry in the industrial community [3], a reliable and purpose designed flow reactor with optimal component matching, easy control over reaction conditions (e.g. temperature, pressure, exposure time, & wavelength of emitted light) and scale-up potential is undeniably essential.

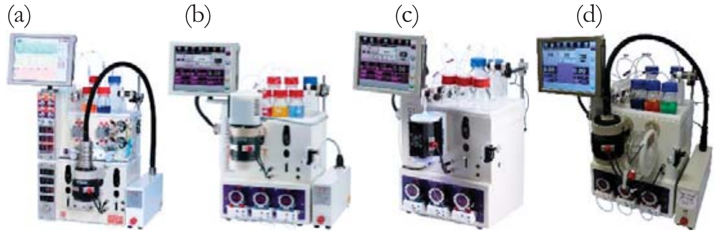
(a) (b) (c)



Figures 1. Examples of improvised flow photoreactors: (a) dual-capillary reactor with internal fluorescent tube, (b) block reactor under UV-panel and (c) capillary tower inserted into chamber photoreactor.

In March 2014, Vapourtec launched its UV-150 photochemical reactor [4]. Since its introduction, additional accessories and features have been continuously released (**Figures 2**), which have made this device family suitable for a wide range of photochemical applications. Subsequently, the systems have been rapidly adopted by the academic and industrial communities and have been featured in over 75 publications [5].

(a) (b) (c) (d)



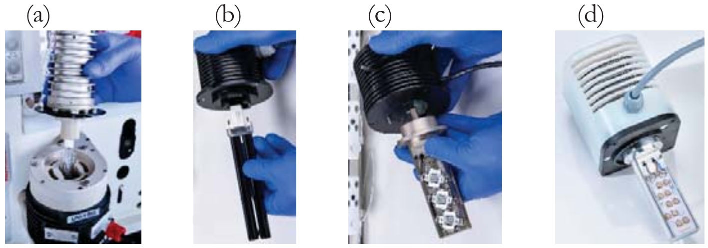
Figures 2. (a) UV-150 reactor R-series, (b) high power LED reactor E-series, (c) immobilized photocatalyst reactor E-series and (d) UV-150 reactor E-series in tandem configuration.

Each photochemical module can be easily plugged into either the E-Series (peristaltic pump) or the R-Series (piston pump) platform. The entire installation of these compact and space-efficient modules and their power supplies takes less than 5 minutes. In tandem configuration (**Figure 2d**), each reactor can be equipped with an additional thermal loop that allows flow photochemical and thermal reactions to be performed in series with precise control over reaction conditions, all from an intuitive touch screen user interface [6].

The UV-150 photochemical reactor chamber can be fitted with three different light sources (**Figures 3**) that cover the entire photochemically important range of 220-650 nm. Each lamp can be changed rapidly by hand within a minute. The available light sources comprise of:

- a dimmable polychromatic (75-150W) medium-pressure mercury lamp,
- low-pressure mercury lamps (9 W) that emit in the UVA, UVB or UVC range, and
- narrowly emitting medium- (60 W) or high-power (150 W) LED panels ranging from 365-525 nm.

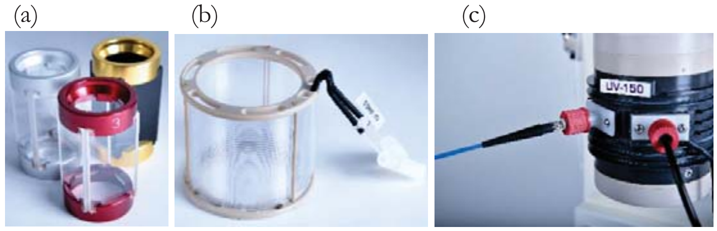
(a) (b) (c) (d)



Figures 3. (a) 150 W medium-pressure lamp, (b) UVA 9 W low-pressure lamp, (c) 60 W LED-array and (d) 150 W high-power LED array.

The medium-pressure mercury lamp can be equipped with a range of easily exchangeable band-pass or cut-off filters (**Figure 4a**) that are placed between the bulb and the reactor coil for a selective wavelength region, and thus permit selective photochemical activation. The reactor cartridge itself consists of a replaceable 2, 5 or 10 mL fluorinated ethylene propylene (FEP) coil with a wall thickness of 0.15 mm and an internal diameter (ID) of 1.30 mm (**Figure 4b**). The coil material is (photo)chemically inert and shows excellent transparency at wavelengths >240 nm. An optional spectrometer can be attached to the reactor housing via a fiber-optic probe (**Figure 4c**), thus allowing real-time monitoring of the transmission spectra. The photoreactor chamber is designed to give safe and accurate heat management. Reactor cartridge and lamp are housed in separate, sealed quartz chambers and air is circulated within each chamber at high flow rates to dissipate heat. An optional external cooling module utilizes a dry-ice filled Dewar that delivers chilled gas (N_2 or compressed air) to the irradiation unit. The reactor temperature can thus be varied with ±1°C precision between -5 and +80°C for the medium-pressure mercury lamp or between -40°C and +80°C for the low-pressure mercury lamps and LED arrays. The temperature is measured directly at the reactor wall for most accurate representation.

(a) (b) (c)



Figures 4. (a) Selection of glass-filter inserts, (b) 10 mL-FEP reactor coil and (c) fiber-optic probe and temperature sensor attachments.

The alternative packed-bed photoreactor system (**Figure 2c**) has been developed for illuminations involving immobilized or solid photocatalysts [7]. It consists of a glass column that allows adjustable packed bed volumes of 0.3-3 mL. The column is surrounded by three LED arrays (60 W) with variable wavelengths of 365-700 nm.

The photoreactor series developed by Vapourtec Ltd has been successfully applied to a wide range of homogeneous and heterogeneous (gas-liquid, solid-liquid or gas-liquid-solid) photoreactions. Optimization of reaction conditions (e.g. concentrations, wavelengths, light power or temperature) is rapidly achieved using small reagent volumes. Scale-up is then simply conducted by continuous operation over longer periods of time using larger stock solutions of reagents. In contrast to other commercial flow devices that are based entirely on LED light-sources, the UV-150 module allows photochemical investigations in the important UVC- and UVB-ranges. The simple interlock design of the individual components also permits an easy modification of the reactor system based on experimental requirements. Likewise, the unique tandem option readily facilitates in-series photochemical-thermal operations in the same continuous-flow in a single compact device ('telescoping') [8].

For more information, application notes and references, please visit https://www.vapourtec.com (last accessed December 20th 2019).

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