



Gas Module - Gaseous reagent organic chemistry makes easy



H-Cube Pro™ and Gas Module

INTRODUCTION

The new Gas Module works seamlessly with the new H-Cube Pro™ allowing a further 13 gases to be used at up to 100 bar using the same touch screen intuitive controls. Reactions such as carbonylation or oxidation can now be performed on the H-Cube Pro™ at the same high pressure and ease of use, extending the reactor's chemistry capacity significantly.

GAS-LIQUID REACTIONS IN CONTINUOUS FLOW

The use of continuous flow tri-phasic, gas-liquid-solid reactions employing immobilized metal catalysts has increased significantly over the past few years. For heterogeneous catalytic gas-liquid reactions the primary advantages of flow result from the high specific interfacial area. The power of this technique is particularly obvious in gas-liquid-solid triphasic reactions involving various gases, a substrate dissolved in a solvent, and an immobilized solid precious metal catalyst. Owing to the large interfacial areas and the short path required for molecular diffusion in the very narrow microchannel space, very efficient gas-liquid-solid interaction occur. In microreactors, reactions could take place which are not attainable in normal batch systems in minutes instead of hours. An additional advantage of flow reactors is that the small volume reduces the damage potential of explosions and therefore conforms with the 12th principle of green chemistry. Another green advantage: using gaseous reagents over chemicals reduces work-up and purification [1].

H-Cube Pro™ Gas Module provides you:

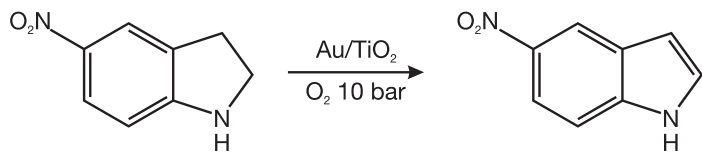
- 13 gases in one device
- Fast reactions - chemistry in minutes
- Powerful: Up to 100 bar capability
- Intuitive User Interface
- Fast: Just like with hydrogenation on H-Cube Pro™
Reactions with other gases can complete in less than 10 minutes
- Robust: All high quality stainless steel parts
- Simple: 3 button stand-alone control or via simple touch screen control on H-Cube Pro™

Gas Name	Gas Tape	Max. pressure (bar)	Min. flow rate (mL/min)	Max. flow rate (mL/min)	Min. flow rate (mmol/min)	Max. flow rate (mmol/min)	Min. flow rate (mg/min)	Max. flow rate (mg/min)
Air	Air	100	1	99	0.04	4.41	1.2	127.5
Argon	Ar	100	1	139	0.04	6.17	1.7	245.9
Ethylene	C ₂ H ₄	100	1	62	0.04	2.74	1.2	76.6
Ethane	C ₂ H ₆	25	1	49	0.04	2.17	1.3	65.1
Methane	CH ₄	100	1	76	0.04	3.38	0.7	53.7
Carbon monoxide	CO	100	1	99	0.04	4.40	1.2	123.2
Hydrogen	H ₂	100	1	100	0.04	4.46	0.1	9
Helium	He	100	1	138	0.04	6.13	0.1	24.7
Nitrogen	N ₂	100	1	100	0.04	4.42	1.2	123.8
Nitrous Oxide	N ₂ O	25	1	75	0.04	3.33	1.9	146
Nitric Oxide	NO	100	1	99	0.04	4.42	1.3	132.6
Oxygen	O ₂	100	1	98	0.04	4.37	1.4	139.9
Synthesis Gas	Syn	100	1	100	0.04	4.43	0.6	61.6

Table 1. Available gas flow rates



1. OXIDATION OF INDOLINE TO INDOLE



Sample (mL)	Lig. flow (mL/min)	O ₂ flow (mL/min)	Temp. (°C)	Conversion (%)
1	1	10	75	8
1	1	10	150	95
8	1	10	150	50
1	0.5	50	150	>98
5	0.5	50	150	>98
5	0.5	50	150	>98

Table 2. Conditions and results of oxidation reaction

EXPERIMENTAL PROTOCOL:

H-Cube Pro™ was coupled with Gas Module and an oxygen cylinder. The following reaction parameters were set before starting the experiment using pure acetone; temperature: 150 °C; liquid flow rate: 0.5 mL/min; gas flow rate: 50 mL/min; catalyst applied: Au/TiO₂ (length of catalyst cartridge: 70 mm, Order No. THS 01639); system pressure: 10 bar. Reactant solution was prepared by dissolving 82 mg 5-nitroindoline (0.5 mmol) in 10 mL acetone. When the “Stable” sign appeared in the status line of the H-Cube Pro™, the inlet valve was switched from “Solvent” to “Reactant”, and the solution of 5-nitroindoline passed through the catalyst cartridge. Collection of the product was started at the “Product” outlet after switching the outlet valve to Product. When all the 10 mL reactant solution was used up, the inlet valve was switched back to Solvent. Product collection was continued for an additional 10 min, in order to recover the total amount of product from the catalyst cartridge. The product solution was analyzed by GC-MS without any further dilution.

Batch reference: Oxidative aromatization using activated carbon molecular oxygen system [2]. Yield: 63 %, 120 °C, 9 h

CONCLUSION

The H-Cube Pro™ Gas Module shows several advantages: the gas insertion is very well regulated for high reproducibility; it could generate high capacity gas flow of various gases with excellent gas mixing which allows the user to perform many gas-liquid-solid reactions in flow that are normally difficult to carry out in batch. The H-Cube Pro™ Gas Module readily increases the chemistry and parameter space making it available for the organic chemistry laboratories.

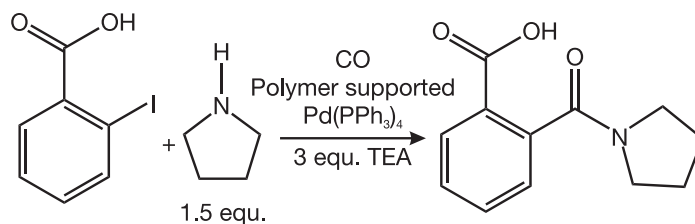
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2. AMINOCARBONYLATION OF IODO BENZOIC ACID WITH PYRROLIDINE



CO flow rate (mL/min)	10	30	30	30	60	60	60	60
Conversion %	60	65	66	62	79	79	79	82

Table 3. Results at various CO flow rates

EXPERIMENTAL PROTOCOL:

H-Cube Pro™ was coupled with Gas Module and a carbon monoxide cylinder. The following reaction parameters were set before starting the experiment using pure acetone; temperature: 100 °C; liquid flow rate: 0.5 mL/min; gas flow rate: 10, 30, 60 mL/min; catalyst applied: Pd(PPh₃)₄ polymer bound (length of catalyst cartridge: 70 mm, Order No. THS 05134); system pressure: 30 bar. Reactant solution was prepared by dissolving 496 mg 2-iodobenzoic acid (2 mmol), 213 mg pyrrolidine (3 mmol) and 607 mg triethylamine (6 mmol) in 200 mL tetrahydrofuran. When the Stable sign appeared in the status line of the H-Cube Pro™, the inlet valve was switched from Solvent to Reactant, and the reaction mixture passed through the catalyst cartridge. Collection of product was started at the Product outlet after switching the outlet valve to Product. When the 10 mL reactant solution was used up, the inlet valve was switched back to Solvent. Product collection was continued for an additional 10 min, in order to recover the total amount of product from the catalyst cartridge. The product solution was analyzed by LC-MS without any further dilution.

REFERENCES

- [1] Irfan, M; Glasnov, T; Kappe, C O; *ChemSusChem*, **4**, 3, 300–316, (2011)
- [2] Nomura, Y; Kawashita, Y; Hayashi, M; *Heterocycles*, **74**, 629 - 635, (2007)

