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Data Acquisition System for Chemical Iodine Generation Suitable for Flowing Medium Chemical Oxygen Iodine Laser

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ABSTRACT

Development of infrared flowing medium lasers needs to be envisaged in a manner that practical aspects such as system compactness, short readiness time, low system size, weight and power are met to make them field deployable. In this context, the critical aspect of in-situ production of lasing species (Iodine) in Chemical Oxygen Iodine Lasers (COIL), one of the most potent flowing medium lasers, has been investigated. The paper dwells on chemical generation of iodine and its precise flow and parameter control by implementing a customised Data Acquisition System (DAS). Iodine is generated in a chemical reaction of Cuprous Iodide (CuI) with chlorine. This is achieved by precisely controlled flow of chlorine diluted with a carrier gas (N_2) in a ratio of 1:2. DAS includes regulated gas feed, accurate thermal stabilisation, relevant diagnostics and implementation of necessary safety interlocks in a real time operation scenario for establishing the system efficacy and scalability. The studies have demonstrated chemically generated iodine flow rate of ~ 1.2 mmol.s^{-1} for Cl_2 flow rate of ~3 mmol.s^{-1} all measured in real time using the developed DAS with a conversion efficiency of 80%. Developed I_2 supply system has potential to deliver iodine on demand with required flow rates, measurement uncertainty of ~ 4.5 percent and advantages of smaller specific weight and size with reduced system readiness time and electrical power supply using DAS system with adequate safety interlocks.

Keywords: Flowing laser; Data acquisition system; Flow rate; Size, weight and Power; SWaP

1. INTRODUCTION

Atomic iodine is the lasing medium in chemical oxygen iodine laser (COIL)[1]. In COIL, the pumping source viz. Singlet Oxygen (SO) is generated by the chemical reaction of chlorine and basic hydrogen peroxide. The singlet oxygen first dissociates the molecular iodine into atomic iodine and then excites the atomic iodine. Several methods for generation of iodine gas such as evaporation, discharge generation methods and chemical methods[1-8] have been reported.

The most conventional method of molecular iodine gas generation for COIL application is the evaporation method[1-4] in which iodine crystals are heated at the controlled temperature range of 80-85° C. However, for scaled up COIL system, the evaporation based iodine generation system become bulky and consumes considerably large amount of electrical power. Apart from being bulky in size, the readiness time of the system is quite large as it requires pre-heating for long duration (few hours for kW level COIL system)[4] and it limits the multiple shot capacity of the laser. Therefore, the evaporation based method is not quite suitable for multi-kilowatt COIL system.

In discharge methods, Iodine gas is obtained by dissociation of various iodine donor compounds (organic iodides, I_2, HI, HI, CH_3I and CF_3I) in the Radio Frequency (RF), Microwave (MW), DC-pulsed or DC-vortex stabilised discharge[5-6]. But, the points of concern are temperature of generator and excess of discharge generator effluents containing various free radicals which are strong quenchers of singlet oxygen.

The molecular iodine can also be generated by the chemical reaction of gaseous Hydrogen Iodide (HI) and Chlorine[7]. The gaseous method has the advantage of better flow control of iodine, however, other gaseous product such as HCl vapours in this method may potentially quench the singlet oxygen thereby diminishing system efficiency. Further, it may also lead to increase in overall mass flow rate of COIL exhaust, which typically demands high mass flow rate of motive gas in active pressure recovery system.

Another chemical method of I_2 generation is based on reaction of solid cuprous iodide (CuI) and gaseous chlorine[8]. *CuI*-Cl_2 reaction method does not produce excess gaseous product or free radicals which are quenchers of singlet oxygen. *CuI* based chemical method also exhibits benefits in terms of small size, reduced weight and low power demand along with diminished system readiness time. Also the iodine flow rate can be accurately controlled by monitoring the chlorine flow rate. *CuI* (in excess amount) reacts with the chlorine and the following reaction takes place:

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$$2CuI + 2Cl_2 \rightarrow 2CuCl_2 + I_2 \quad (1)$$

Since reaction (1) is an exothermic one, temperature may be maintained without any external heating source.

In case of COIL sources, both the medium gain[2-3] and laser output power are strong function of iodine flow rate. Thus, for efficient COIL operation, it is imperative to accurately measure I_2 flow rate and also achieve precise flow control by parametrically studying and establishing optimum operating conditions.

This paper focuses primarily upon chemical generation of iodine by employing CuI-Cl_2 reaction mechanism and performs parametric investigation and analysis by using developed DAS based on wireless Wi-Fi interface[9-14]. DAS is configured in order to specifically meet the needs of online parametric measurement and operational requirements. The study enables to compare the plausible size/weight reduction, diminished electrical power requirements and curtailed system readiness time of the COIL system based on CuI-Cl_2 method of iodine generation against conventional COIL systems using evaporation method for iodine generation.

2. EXPERIMENTAL SETUP

CuI is a photosensitive chemical and is thereby reactive to light. Thus, the reaction chamber is developed form an opaque material such as brass also keeping in view of its compatibility with the constituent chemicals and the products of the reaction. The chamber comprises of 9 guiding vanes (brass) with size of *100mm (l) x 50mm (w) x 3mm (t)* each fixed such that they not only hold the *CuI* powder but also provide the requisite contact surface area along with increasing the overall traverse path length for chlorine gas, thereby increasing the gas residence time. The schematic along with the developed hardware is shown in Figs. 1(a) and 1(b), respectively. The developed reaction chamber has an overall dimension of *200 mm (l) x 120 mm (w) x 50 mm (h)*. The flow cross section between the guiding vanes is *50 mm (h) x 17 mm (w)*. The reaction chamber has necessary provision for installing pressure and temperature sensors for real-time monitoring of the same.

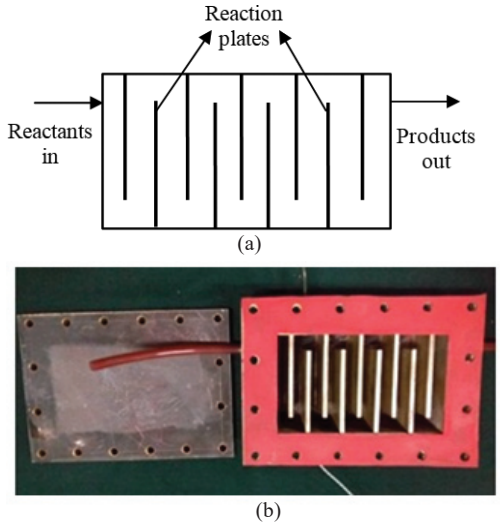


Figure 1. (a) Schematic of reaction chamber and (b) Developed hardware.

The block diagram showing the various elements of chemical iodine generation system is shown in Fig. 2. It consists of a buffer (N_2) gas feed regulation system, pre-heaters for preventing I_2 solidification, transport line heaters, belt heaters for CuI powder bed, I_2 diagnostics cell and finally the effluent neutralizer.

Preheated N_2 and Cl_2 gas mixture is fed to the reaction chamber at controlled pressure and flow rates. The mass flow rates are controlled using the orifice under choke flow condition. Cl_2 gas reacts with the CuI crystals in the reaction chamber leading to production of I_2 gas. The chemically generated iodine is passed through a diagnostic cell and the concentration is measured using non intrusive optical technique. Finally, the residuals consisting of I_2 and Cl_2 gas with buffer N_2 are passed through a chamber of KOH solution for neutralisation of the halogen constituents.

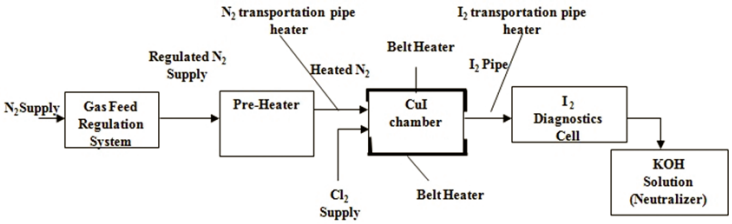


Figure 2. Block diagram of CuI based chemical iodine generation system.

3. CUSTOMISED DATA ACQUISITION SYSTEM (DAS)

Generically, the accurate characterisation of the chemical iodine generation system coupled with corresponding parametric investigation demands that real-time measurement, control, display and storage of operation parameters such as flow rates of constituent gases and buffer gas, gas supply temperature, chamber and heater temperatures. All these operations to be carried out together with accurate determination of I_2 produced employing appropriate diagnostics. Hence, in order to fulfill all of the above mentioned functions in real-time, the hardware needs to be suitably interfaced with a dedicated 16 bit resolution and varying sampling rates (12.5 Samples/s/channel to 25 kSamples/s/channel Data Acquisition System (DAS).

The architecture of the developed DAS is shown in Fig. 3. It consists of a Sensor system, Acquisition and control part, Transceiver (EKI-1361), Processor controller ARK-1122C (slave unit), router D-Link wireless AC750 and a display and control device with application software (master controller). Slave ARK -1122C unit is a processor which acquires data from the sensors via DAS card (ADAM 3014 and USB 4716) and controls the whole process of the operations, but only after receiving command from the master controller. Router is D-Link wireless AC750 dual band router with 750 Mbps wireless data transfer speed. It provides security with advanced WPA2™ encryption and easy secure connection using Wi-Fi protected setup™ (WPS). In DAS, EKI-1361is used as a serial server and performs both as transmitter and receiver. ARK-1122C is used for configuring fast sampling rate channels. Both these cards process and control the signals

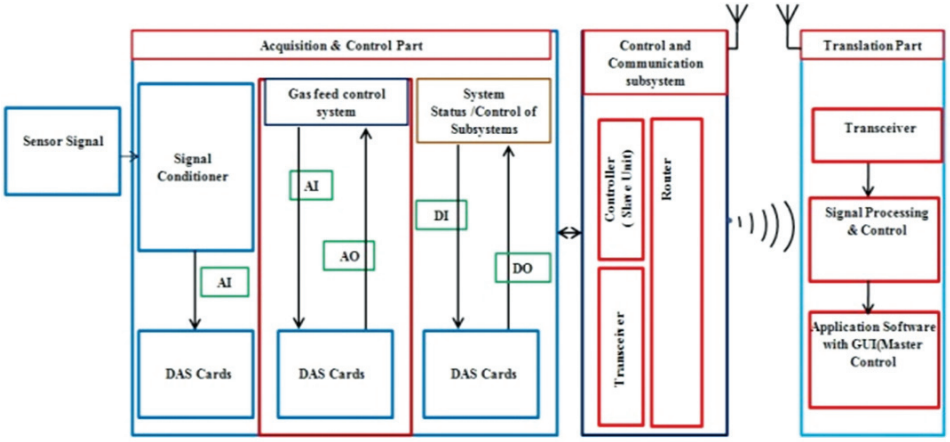


Figure 3. DAS architecture.

from Advantech cards such as Model No. ADAMs 4117, 4118, 4015, 4017, 4051 and 4069. Wireless serial server card EKI-1361 and ARK-1122C are based on Wi-Fi 802.11b/g/n. Wireless communication eliminates the need for physical connection between control and communication subsystem and translation part (transceiver, signal processing and control and master controller). Experiments can be performed from 35 m with obstacles like concrete walls, trees etc. and from 80 m without any obstacles using wireless interface between slave unit and master controller.

Figure 4 shows DAS interfacing with experimental setup of CuI based chemical iodine generation system and its constituent elements for precise control, monitoring, storage and display of influencing reaction parameter. The developed DAS interface performs the following functions remotely:

- Controlling (Digital Output, DO), monitoring (Digital Input, DI) and acquiring (Analog Input, AI) temperatures at various locations (i.e. pre-heater, N_2 transport line, CuI bed, I_2 transport line)
- Controlling (Analog Output, AO), monitoring (DI) and acquiring (AI) pressures at various locations, which in turn control flow rates
- Controlling (AO) and acquiring (AI) flow rates in chlorine, iodine and nitrogen lines
- Online status (on/off) display of various heaters (DI)
- Digital control of dc current operated (~500 mA) electrical pressure reducer (EPR) and solenoid valve (SV) using ADAM 4069 (power relay module) with Modbus protocol and ADAM 4017. Iodine is generated due to the sequential operation of the valves in a predefined manner
- Displaying of current status (DI) of SV using ADAM 4051
- Acquisition of iodine diagnostic signal (AI)
- Proper sequencing of chlorine and nitrogen supply
- Incorporating safety aspects in the chlorine gas supply line due to toxic nature of chlorine.

Both Chlorine (Cl_2) and Nitrogen (N_2) pressure were measured using pressure sensors (Xian Yunyi Instruments Pvt. Ltd., China). The temperature generated in the CuI during the exothermic reaction between CuI and chlorine is measured with the help of K type thermocouple- response time: 20ms, accuracy: ±0.5% (of FSR), and linearity in the operating range, Company: RS Controls India Ltd. Stock No. 6212170.

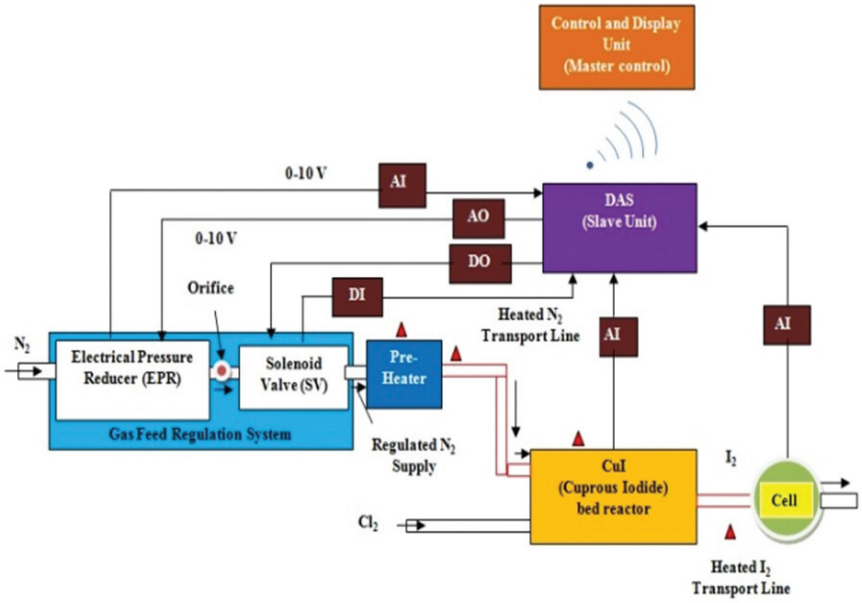


Figure 4. DAS interface for chemical generation of I_2.

The most critical aspects as far as DAS is concerned are viz., *precise flow control, thermal stabilisation, iodine diagnostics and safety interlocks.*

3.1 Gas Flow Control

The performance of the CuI based chemical generation of iodine scheme depends strongly on the flow rates of the nitrogen and chlorine gases. N_2 and Cl_2 gases are controlled using DAS in 2:1 ratio and fed into CuI bed for successful generation of iodine. Control of gases line pressure is achieved using an EPR (electrical pressure reducer) by varying its analog input voltage (0–10 V) from the DAS. EPR (SMC make, ITV series) also provides 0-10 V output to the DAS (AI) for analysis of system performance. Solenoid valve (SMC Make, VXZ series) operated using 24 V DC controls the sequence of gas supply into the system preset using DAS sequence control. DAS also has an inbuilt feedback to continuously monitor the real time status (on/off) of solenoid valve obtained using a digital input (DI) during the duration of system operation.

The real-time parameters such as temperature, pressure at different locations of reaction chamber, gas supply lines, pressure reducers and also the photo-detector signals are fed to the analog input channels of ADAM 3014, USB 4716 and ARK 1122C with accuracies of ±0.5%, ±0.1%, ±0.2% and ±0.5% respectively. Graphical user interfaces (GUIs) were developed using Lab VIEW software for control and depiction of all relevant parameters. A representative GUI window for flow control of gases is shown in Fig. 5.

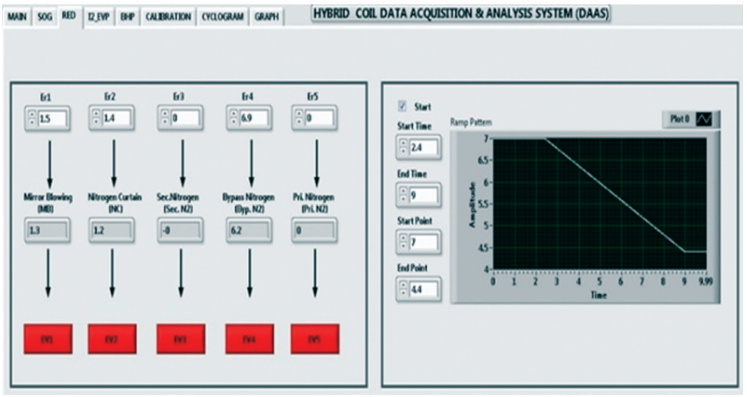


Figure 5. Graphical user interface for gas flow control.

3.2 Thermal Stabilisation

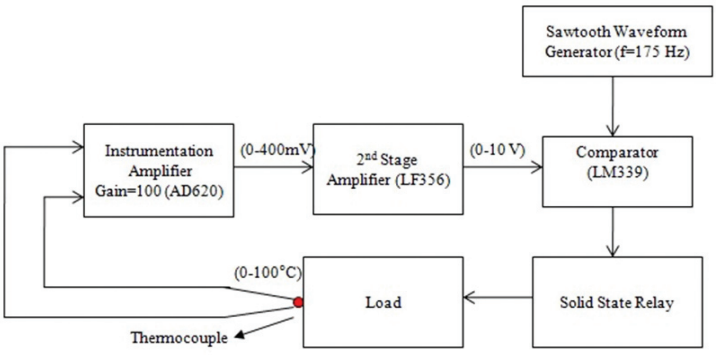
The continuous monitoring and accurate heater control is imperative for reproducibility of iodine flow rates. This is accomplished using developed thermal stabilisation scheme as shown in Fig. 6(a). The scheme utilises a pulse width modulation based technique to maintain the temperature of the transport lines of the system within 55-75°C with an accuracy of ±0.5°C. Pulse width modulation (PWM) enables reduction in overall energy consumption of the module. The temperature controller circuit [Fig. 6(b)] has been developed using LF 356, AD 620 instrumentation amplifier (IA), LM 339 comparator ICs with a thermocouple (T/C) input and a proportional control output. The gain of the amplifier module is~ 2500. By comparing amplifier output signal with the saw tooth waveform, pulse-width modulated output is generated which is used for on/off control of heaters.

3.3 Safety Interlocks

The operation of the CuI based iodine generation system involves hazardous chlorine and iodine gases. Toxic chlorine has adverse effects on human health such as irritation of eyes, mucous membranes and respiratory system leading to potential breathing problems along with producing pale, cold and clammy skin[15]. Both concentration and exposure time are of concern in handling of these gases and needed to be taken into account for prescribing the exposure limits. Iodine has similar side effects apart from also having an added influence on human nervous system and affecting organs without direct blood supply. The exposure limits for chlorine and iodine are listed in Table 1. Hence safety interlocks are essentially required for safe operation.

Table 1. Exposure limits for chlorine and iodine[15]

Exposure Limit	Chlorine (ppm)	Exposure Limit	Iodine (g)
Allowable limit for prolonged exposure (max)	1	Probable lethal dose of free iodine	2-4
Allowable exposure limit for ½ to 1 h (max)	4		
Throat irritation (min)	15.1		
Coughing irritation (min)	30.2		



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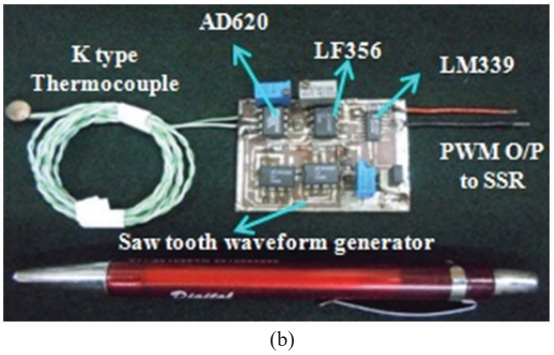


(b)

Figure 6. (a) Thermal stabilisation scheme block diagram and (b) Developed hardware.

The safety control system primarily consists of an electrochemical sensor (Make: Calibration Technologies Inc, Columbia) for both the halogen gases having an accuracy of ±0.5% (of Full Scale Range) and long term stability is lesser than 0.1% FS/month. The sensor generates a 4-20 mA signal corresponding to the leak concentration. The current signal is converted into a voltage signal by using current to voltage converter. A reference signal or the threshold signal is also fed to the comparator (LM339), which compares both the acquired and reference signals and in case the threshold is exceeded a command to activate the solid state relay (Make: ERI, model no.: 001 JDA 332500, Input: 3-32 V dc, Output: 220 V ac, 25 A) in case of leakage is issued. The interlocking scheme activates the auto warning safety control system, which issues a warning signal and an audible alarm to sensitise the personnel at work and simultaneously a system shut off command is initiated. The interfacing scheme is also shown in Fig. 7.

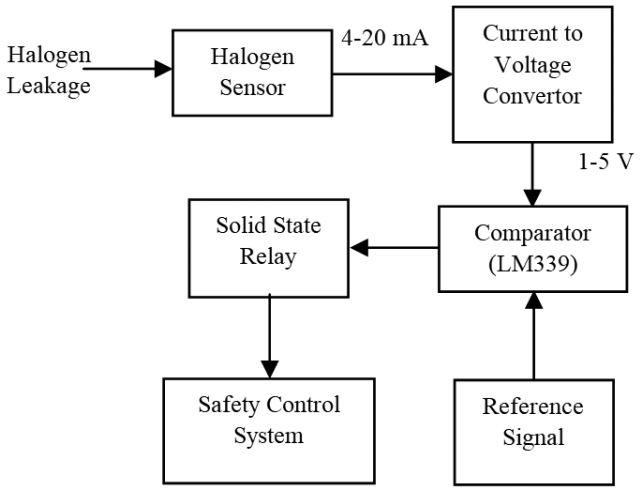


Figure 7. Block diagram of auto warning safety control system.

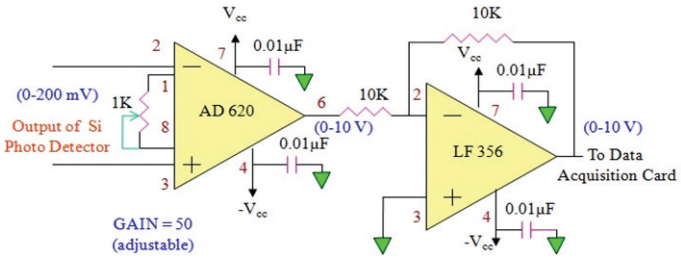
3.4 Iodine Diagnostics

Optical diagnostics based on absorption spectroscopy has been utilised for iodine concentration measurement[16-18] using DAS. The transmitted light (without and with iodine) mostly at peak iodine absorption wavelength of 490 nm is passed through a probe cell of nearly 5 cm length and focused on to a suitable Si-detector.

Since the detector output signal is typically low, an amplifier interface electronic circuit [Fig. 8(a)] has been designed and developed for iodine concentration measurement to provide 0-10 V output corresponding to the detector signal 0-200 mV using amplifier (AD 620, low noise instrumentation amplifier) and Field Effect Transistor (FET) based IC LF356 (Gain = 1, high CMRR). Developed amplifier interface circuit and silicon photodiode detector (RS Stock no 303674, active area 100 mm^2) are assembled in a single unit [Fig. 8(b)].

4. RESULTS AND DISCUSSION

Figure 9(a) depicts the developed system hardware for chemical iodine generation. In order to establish the necessity of pre-heaters, few initial experiments were carried out by supplying nitrogen directly (i.e. without heating the transport lines) with chlorine for dilution without using them.



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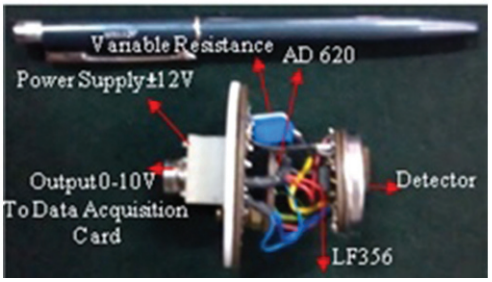
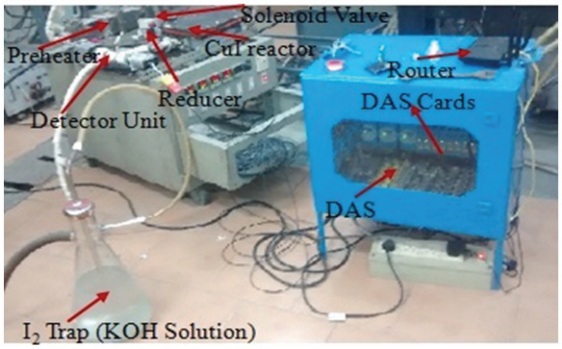


(b)

Figure 8. (a) Amplifier circuit and (b) Developed amplifier interfaced with detector.



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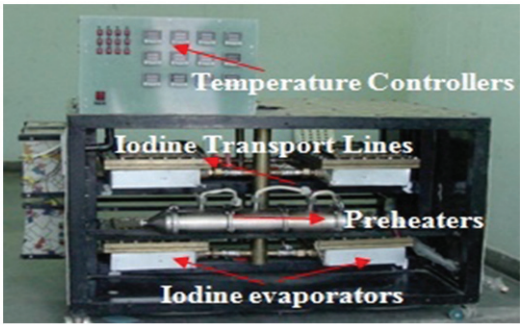


(b)

Figure 9. Experimental set-up for (a) Chemical method and (b) Evaporation method.

It was observed that although iodine was generated, it largely precipitated in the downstream region of the reaction chamber mainly in the iodine diagnostic cell and the effluent discharge lines. Hence, a pre-heater was incorporated into the sequence to provide heated nitrogen (55-75°C) to the CuI chamber. Also, in initial optimisation, a flask cooled with liquid nitrogen ethylene glycol mixture was employed to precipitate the iodine before the remaining effluents exhausted to the scrubber. The difference between the final and initial weight of the flask provided a reasonable estimate of the iodine generated in the

reaction for the duration of the experiment and was compared with results of the iodine diagnostics.

Also, the corresponding system hardware for evaporation based iodine generation is also shown as Fig. 9(b) for comparison. In evaporation method, iodine is used in its basic form i.e. in crystal form. Iodine crystals are heated in the evaporator system in order to maintain the temperature at around 75°C for generating iodine molecules required for COIL operation. But this method requires a large evaporator system, more time to get uniform iodine, several heaters of high electrical power (in kW) at different locations in evaporator system in order to avoid solid iodine precipitation. Electricity consumption increases as we tend to high power level of COIL. On the other hand, developed chemical scheme has much smaller heating load. The is mostly because only the temperature of the transport lines and CuI bed is to be maintained through a feedback mechanism within 55-75°C in order to avoid in-situ precipitation of iodine. Relative combined std. uncertainties in measurement of temperature, pressure, Electrical pressure reducers and photo-detector signals during experiments are 5.8%, 0.3%, 2.3% and 5% respectively.

The comparative heat loading for the indicated iodine flow rates is detailed in Table 2.

It is also apparent from Fig. 9 that the size and weight of chemical based system is significantly lower than the evaporation based iodine generation system. The comparative Size, weight and power (SWaP) estimates per kW of COIL laser power are summarised in the Table 3.

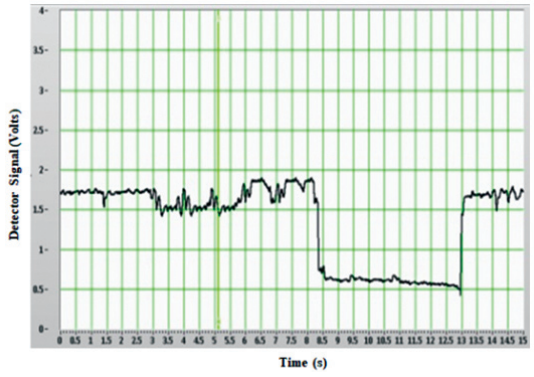
Table 2. Heating requirements

CuI based chemical method		Evaporation method	
Heater	**Wattage (W)**	**Heater**	**Wattage (W)**
Pre-Heater	200	Pre-Heater	1250
N_2 Transportation Pipe Heater	150	N_2 Transportation Pipe Heater	500
CuI chamber Belt Heater	600	Iodine crystal chamber	500
I_2 Transportation Pipe Heater	400	I_2 Transportation Pipe Heater	750
Total Power Requirement	1350 W	Total Power Requirement	3000 W

Figures 10 and 11 show graphical comparison of the detector (Make: Centronic, RS Controls India Ltd., Stock no: 303674, Spectral sensitivity : 0.5 A/W) signals (volts) and iodine flow rates (mmole/s) recorded for chemical and conventional iodine generation methods using the dedicated DAS. Iodine molar flow rate is expressed using the Beer-Lambert law and the perfect gas law equation,

$$\dot{m}_{I_2} = \left\{\frac{M_c k T_0}{\sigma_v L}\right\} \frac{\ln\left(\frac{I_0}{I_v}\right)}{P_{tot} - \left(\frac{kT_0}{\sigma_v L}\right)\ln\left(\frac{I_0}{I_v}\right)} \quad (2)$$

where I_0 and I_v represent light intensity without and with iodine (Fig. 10) and obtained in the form of iodine detector signal, σ_v – iodine absorption cross section (2.1×10^{-22} m^2) at 490 nm, L-length of the iodine cell unit, k-Boltzmann constant, T-gas temperature, P_{tot} -measured total pressure, M_c -N_2 molar flow rate.



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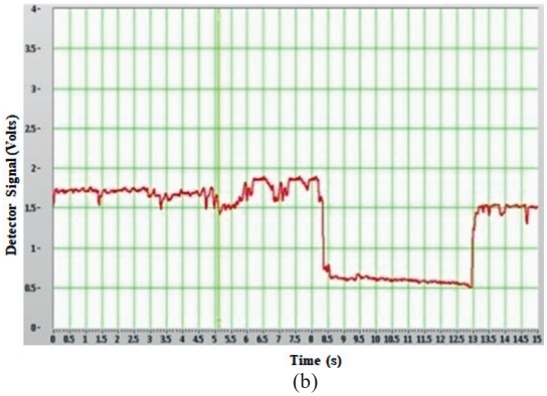

(b)

Figure 10. Obtained detector signal (V) vs. Time (s) for (a) Chemical and (b) Evaporation method.

Table 3. Comparison of Chemical and Evaporation methods for iodine generation

Parameters (per kW laser power)	Chemical generation method	Evaporation method	Inferences
Iodine flow rates (mmol.s^{-1})	0.6-0.7	0.6-0.7	Exp. Achieved values
Size (ft^3)	0.5	1.5	66.7% reduction
Weight (kg)	3.5	7.3	52.1% reduction
Power requirement (kW)	0.675	1.5	55% reduction
Readiness time	Nil	~ 1hr	Iodine on demand in the chemical method
Multiple firings	Minor SWaP penalty	Major SWaP penalty	Only larger stock of solid CuI reagent is required in the Chemical method

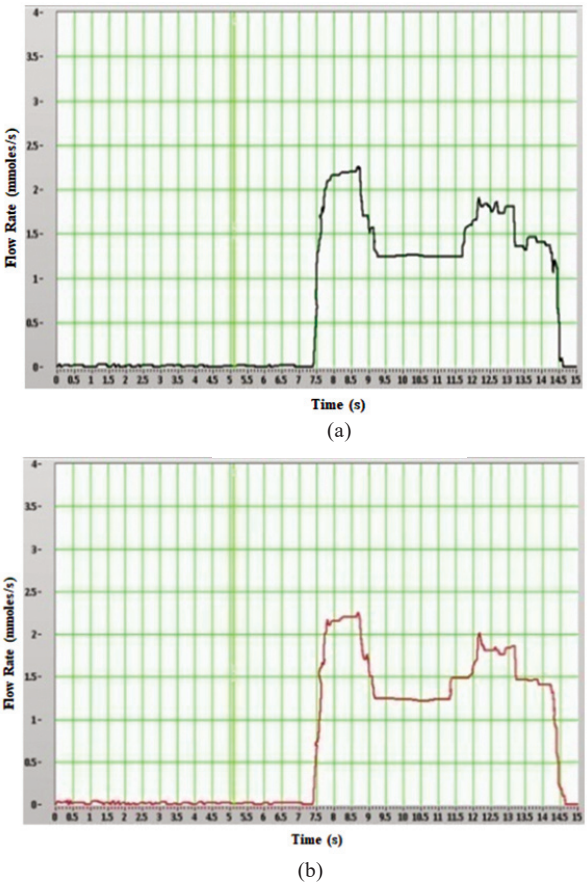

Figure 11. Temporal variation-Iodine flow rate (mmole/s) vs. Time (s) in (a) Chemical and (b) Evaporation method.

The combined uncertainty in iodine measurement is obtained from the sensitivity calculation by applying partial differentiation fraction in equation (2) and is given by,

$$u\left(\dot{m}_{I_2}\right)=\sqrt{\begin{array}{l}\left(\frac{\partial \dot{m}_{I_2}}{\partial M_c}u\left(M_c\right)\right)^2+\left(\frac{\partial \dot{m}_{I_2}}{\partial T_0}u\left(T_0\right)\right)^2+\\ \left(\frac{\partial \dot{m}_{I_2}}{\partial L}u\left(L\right)\right)^2+\left(\frac{\partial \dot{m}_{I_2}}{\partial P_{tot}}u\left(P_{tot}\right)\right)^2+\\ \left(\frac{\partial \dot{m}_{I_2}}{\partial I_0}u\left(I_0\right)\right)^2+\left(\frac{\partial \dot{m}_{I_2}}{\partial I_v}u\left(I_v\right)\right)^2\end{array}} \quad (3)$$

It is evident that iodine has been generated successfully by employing the chemical method and obtained iodine flow rate is ~1.2 mmole.s^{-1} for Cl_2 flow rate of ~3 mmole.s^{-1} with a conversion efficiency of nearly~ 80% and almost same laser power is generated (Fig. 12). Uncertainties in iodine and chlorine flow rates measurement are ~4.5% and ~3.5% respectively. The chemical generation of iodine is beneficial in terms of realisation of practical and field deployable COIL systems capable of having a smaller SWaP footprint together with significantly lower system readiness and turnaround time with effective multiple firing capabilities.

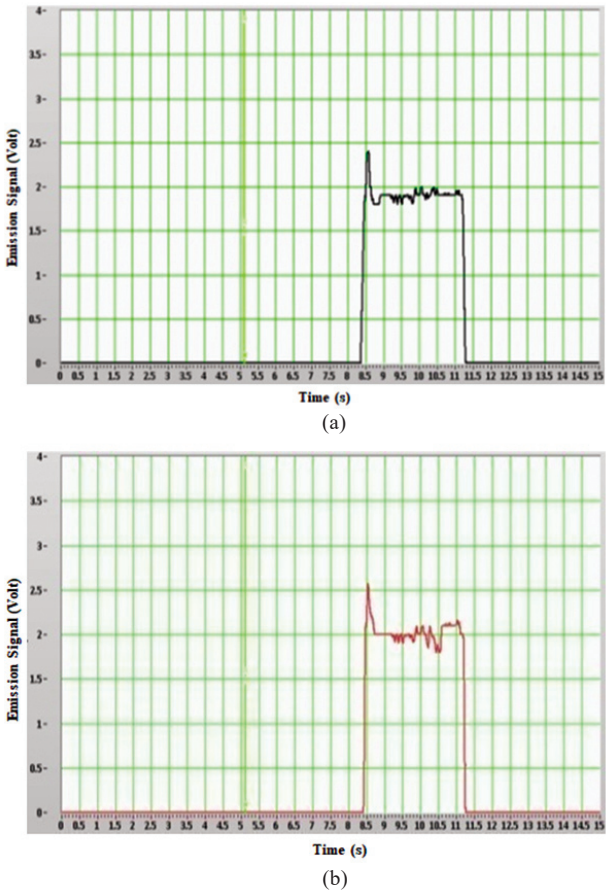

Figure 12. Temporal variation-Emission signal (V) vs. Time (s) in (a) Chemical and (b) Evaporation method.

5. CONCLUSION

A custom Data Acquisition System (DAS) for proving the effectiveness of chemical generation of iodine for COIL systems has been developed. The interfaced DAS has enabled safe operation and parametric system optimisation by incorporating necessary provisions of precise flow control, thermal stabilisation, complex optical diagnostics and safety interlocks using pre-sequenced time controls. DAS has enabled proving the efficacy of chemical iodine generation methodology based on CuI-Cl_2 reaction dynamics. Typical iodine flow rate observed are ~1.2 mmole.s^{-1} for Cl_2 flow rate of ~3 mmole.s^{-1}. From Eq. (1), generated Iodine flow rate must be ideally ~1.5 mmole.s^{-1} from 3 mmole.s^{-1} of Chlorine. Hence, the conversion efficiency of our system is {(1.2/1.5)*100} = 80% for an optimum buffer dilution of 2:1. The performance of COIL source remains unaffected with alteration iodine supply system from evaporation based to chemical based. However, the chemical methods offers lucrative benefits in terms of significant savings in terms of overall system SWaP requirements and providing iodine on demand (with measurement uncertainty of ~ 4.5 percent) , thereby negating the need for lead time typically required for COIL firing. These savings may prove to be crucial for envisaging high power field deployable practical COIL systems with high overall technical efficiency (Laser power to System weight, kW/Kg).

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