Newcastle University
School of Chemical Engineering & Advanced Materials

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pig MONTHLY

process intensification group | www.pig.ncl.ac.uk | www.pinetwork.org

In This Issue

HEXAG and PIN Meetings

HEXAG (Heat Exchanger Action Group) and PIN (Process Intensification Network) meetings were held on 20th & 21st June in Merz Court. Both meetings attracted about 50 members from Germany, Belgium, Finland, The Netherlands, Norway, Spain and Russia, as well as the UK. It is worth noting that PIN and HEXAG network meetings now total well over 50 to date – and we boast a global membership. HEXAG and PIN are co-sponsors of the UK Heat Transfer Conference at Brunel University in September.

A strong theme of HEXAG this year was the heat pipe, with applications such as nuclear waste pond thermal control (one feature of the talk by IKE in Stuttgart who pioneered work on heat pipes over 50 years ago) and satellite heat transfer, an area in which Aavid Thermacore in Northumberland is increasingly competitive. Additive manufacturing of heat exchangers is a growth area and HiETA, with whom we collaborate in the aerospace sector, gave us an excellent insight into the capabilities in manufacture not feasible using other fabrication methods.

After a dinner at the Pitcher and Piano on the Quayside where HEXAG and PIN intermingled, the following day was devoted to process intensification, with the EC IbD Project Manager from IRIS in Barcelona giving an overview of the activities. Johnson Matthey and Huntsman were among the industrial organisations presenting, and our PI Group made several contributions.

Alex Koblov from SIBUR, the largest petrochemical company in Russia (based in Tomsk) gave short talks at both meetings, and we are hoping to run a course on PI in Russia for their staff, as well as become involved in R&D on their behalf.

Some of the presentations at both meetings will be on the respective web sites in a week or two – see www.hexag.org and www.pinetwork.org.

David Reay

Upcoming Conferences

- **15th UK Heat Transfer Conference** (4-5 Sep 2017, Brunel University, London)
- ***WCCE10+ECCE11+ECAB4+IPIC*** (1-5 Oct 2017, Barcelona, Spain). Late Registration Deadline: 20th September
- **Global Conference on Catalysis and Reaction Engineering (GCR-2017)** (19-21 Oct 2017, Las Vegas, US). Final Abstract Submission: 26th August 2017
- **2017 *AIChE* Annual Meeting** (Oct 29-Nov 3 2017, Minneapolis, US). Early Registration Deadline: 18th September
- **Green Materials & Technology Conference** (4-8 Feb 2018, Singapore). Abstract Submission Currently Open
- **ChemEngDayUK2018** (27-28 March 2018, Leeds, UK). Abstract Submission Opening: September 2017
- **9th International Conference on Biofuels and Bioenergy** (29-30 March 2018, Edinburgh, UK). Abstract Submission Opening: Currently Open
- **2018 *AIChE* Spring Meeting** (22-26 April 2018, Orlando, Florida). Abstract Submission Deadline: 13 November 2017
- **2nd International Biotechnology Congress (IBC-2018)** (25-27 April 2018, Dalian, China). Abstract Submission Deadline: 20 February 2018
- **25th International Symposium on Chemical Reaction Engineering, ISCRE25** (20-23 May 2018, Florence, Italy). Abstract Submission Deadline: October 2017

CAMURE-10, Qingdao, China

Steven recently attended the 10th International Symposium on Catalysis in Multiphase Reactors and Multifunctional Reactors (*CAMURE-10 & ISMR-9*) held in Qingdao, China from July 7th—10th. The main theme of the conference was "Advanced Catalysis and Green Chemical Reactor Engineering for Sustainable Process Industries". Steven presented a Keynote speech ("Surprises in a Conventional Stirred Reactor: Theory and Applications") and also chaired the session "Stirred Reactor & Mixing" with Prof David Agar (TU Dortmund).

Steven presenting a keynote speech (L) and chairing a session (R) at CAMURE-10

UK Algae Congress, Swansea

Muayad and Musa gave oral presentations at the 7th UK Algae Congress held in Swansea (from 6th—7th July). Themes of the meeting included: algae circular economy, algal biotechnology, disease and contamination, culture & downstream processing, algae economy and algae biochemistry. The talks given by the PI group were titled:

- Drainage enhancement in the continuous foam column used for microalgae biomass recovery | Muayad Abed Alkarawi
- Oil extraction and algae (*Chlorella vulgaris*) separation in a foam column by adding methanol | Salihu Danlami Musa

Group photo taken at the 7th UK Algae Congress

SET 2017, Bologna, Italy

Prof Saffa Riffat, Co-Chair, from Nottingham University addressing the SET Plenary Session

The SET 2017 Conference, held at the University of Bologna, the oldest university in Europe, took our trio to Italy on what may prove to be the warmest week of the year – 40^{o}C the day we flew home! SET (Sustainable Energy Technologies) takes place annually in exotic and interesting places (Ahmad Mustaffar and I attended one in Geneva some three years ago), and while the bias tends to be on the built environment, thermodynamic cycles and infrastructure, it did prove to be a useful venue for presenting data on the EC-funded IbD project. Our paper, authored by David Reay, Colin Ramshaw (Emeritus Professor), Richard Law and Ahmad Mustaffar, brought the 'Intensified by Design' project to a more general audience who we felt could benefit by concentrating upon the means for overcoming fouling in plant – hence the title: "*The Fear of Fouling and Solid Streams – Overcoming this Barrier to Improved Energy Efficiency in Intensified Process Plant*".

In streams and unit operations where solid particles may be present or anticipated most process engineers recognise that they can lead to fouling, ultimately blocking heat transfer channels and plant failure. Other difficulties may be associated with abrasion, separation challenges and particle size classifcation. Introducing the adjective 'smaller' into the definition immediately – for a process engineer – flashes a 'caution' warning. The implied size reduction in plant is the principal feature of 'process intensification', a familiar example of which is a compact heat exchanger with, for example, 2 mm diameter tubular flow passages.

It is in this context, in addition to the other benefits that might arise from the use of PI, that the HORIZON 2020 project 'Intensified by Design' (IbD) led by IRIS in Barcelona has been conceived to address the challenge of solids handling. A major part of the Euro10 million project involves demonstrating, in pilot plant or at full scale, the energy, environmental and economic benefits of PI in sectors including metal ore processing (using flash flotation), ceramic powder drying and granulation, including the use of toroidal fluidised bed technology, and microwave-assisted drying of pharmaceuticals. The Coflore chemical reactor forms the basis of a Case Study specifically dealing with solid particles, and the technique may have wider applications. Another Case Study in Italy involves intensifying metallic powder production using highly efficient classification methods.

We presented information on some of the intensified technologies that might be applied to the sectors targeted in the IbD study, which include pharmaceuticals, mineral and metal processing, ceramics and chemical reactions in the presence of solids, and the likely energy and environmental benefits.

Time out in Venice sipping Prosecco was a highlight of the conference!

Elsevier, a co-sponsor of SET, also used the opportunity to launch the journal '*Thermal Science and Engineering Progress'*, making copies of the first two issues available to all attending and hosting an Editorial Advisory Board meeting – David Reay is Editor-in-Chief, Barbara Sturm, (now at Kassel University) Associate Editor and both Richard and Ahmad are among the 25 EAB members. The first article went on line in mid-March this year, and to date there have been 20,000 downloads.

David Reay

Collaboration with NC State

Two PhD students from the group (Abbas and Warm) are currently working in the US as part of a collaboration between the School of Engineering (Newcastle University) and the Prof Flickinger Group, Dept of Chemical and Biomolecular Engineering at the North Carolina State University. The aim is to "*formulate a highly reactive desiccation tolerant biocomposite for CO_2 mitigation from micro algae using paper, cotton and the loofah*". This involves the adhesion screening and testing of various biopolymer matrices developed from these materials using different types of binders and micro algae cells. Abbas and Warm arrived in the US in June and will return to Newcastle in August. Those involved in the project are:

- Prof Michael Flickinger (Supervisor) and Adam Wallace (North Carolina State University)
- Dr Jonathan Lee (Supervisor), Abbas Umar and Warm In Na (Newcastle University)

PIG News

- Prof Adam Harvey will cease to be the Head of School for Chemical Engineering and Advanced Materials on 31st July, and will be dedicating more of his time to improving the PI group. Any ideas on how to improve the operation/funding/sociability of this group are welcome (email him directly)
- Congratulations to Prof Adam Harvey and Dr Kui Zhang, who have been awarded an EPSRC Impact Acceleration Award worth £75,000
- Dr Kamelia Boodhoo gave a talk on 21st July to International Paints/Akzo Nobel representatives titled "Process Intensification for Scalable Sustainable Processing"
- Prof Adam Harvey visited CPI's National Biologics Manufacturing Centre, Darlington, to investigate potential future collaborations
- Dr Ana Lopez visited Nottingham University recently to learn more about microwave processing

AIChE Spring Meeting: PI Topic Announced

The upcoming Spring Meeting of AIChE (22-26 April 2018, Orlando, Florida) currently has process intensification listed as one of the main topics (Topic C). The deadline for abstract submission is 13 Nov 2017.

New Group Photo

Other Information

- Full contact details and research profiles for the PI group members can be found at the website: www.pig.ncl.ac.uk
- For enquires about collaborations or PhD study, see the website: www.pig.ncl.ac.uk
- If anyone would like to contribute any articles, or if anyone has any ideas regarding the newsletter please contact Jonathan McDonough: jonathan.mcdonough@ncl.ac.uk

New Publications

- Cheng D, Wang S, Kuipers JAM. Modelling study of gas-liquid mass transfer enhancement by cylindrical catalyst particles. *Chemical Engineering Science* 2017, 160, 80-84
- Cheng D, Wang S, Yang C, Mao ZS. Numerical Simulation of Turbulent Flow and Mixing in Gas-Liquid-Liquid Stirred Tanks. *Industrial & Engineering Chemistry Research* 2017, doi: 10.1021/acs.iecr.7b01327, in press (special issue: Prof. Tapio Salmi Festchrift)
- Feng R., Ramchandani S., Ramalingam B., Tan S.W.B., Li C., Teoh S.K., Boodhoo K., Sharratt P. Intensification of Continuous Ortho-Lithiation at Ambient Conditions—Process Understanding and Assessment of Sustainability Benefits. *Org. Process Res. Dev.* (2017) 10.1021/acs.oprd.7b00142
- Ohmura N, Masuda H, Wang S. Intensification of Mixing Processes with Complex Fluids. *Journal of Chemical Engineering of Japan* 2017. In Press
- Wang Y, Xu J, Wang S, Yang C. Quantitive relationship between fluid inhomogeneities and flow enhancement in nanotubes. *Nanoscale* 2017, 9, 6777-6782
- Zivkovic V., Yang H., Zheng G., Biggs M. Time-resolved granular dynamics of a rotating drum in a slumping regime as revealed by speckle visibility spectroscopy. *In: Powders and Grains 2017—8th International Conference on Micromechanics on Granular Media*. Montpellier, France

Recent PIG Seminars

- Ahmed Al-Hatrooshi. Marine Waste Biorefinery. 2nd June
- Dr Ahmad Mustaffar. Twin Screw Granulator for use in the Pharmaceuticals Industry (IbD). 9th June
- Faisal Saleem. Non Thermal Plasma Assisted Hydrocracking of Tar Analogue in Dielectric Barrier Discharge Reactor. 16th June
- Prof Hyoungsoo Kim (Guest Speaker). Fluid and interface problems in Tokamak, oil and alcohol. 30th June
- Tobechi Okoroafor. The sustainability of using carbon dioxide in microbial electrosynthesis. 7th July
- Muayad Al-Karawi. Bubble-microalgae collection probability and flotation kinetics in foam flotation column. 14th July (*see this issue's research spotlight article for more information*)

Harvesting and treatment of microalgae using foam column

Muayad Alkarawi, Musa Salihu, Abbas Umar, Dr Jonathan Lee

Attention has been given recently to move from conventional fuels to renewable-based fuels due to concerns about the sustainability of fossil fuels, fluctuating oil prices, environmental pollution, and global climate change. Microalgae have the potential to play a vital role in the biofuels market. However, high processing costs (harvesting, dewatering, and drying) hinder the production of algal-based biofuel at a competitive price. Harvesting, for example, comprises ~20-30% of the total production cost, necessitating the development of cost effective and reliable bulk harvesting techniques. The challenges of harvesting algae cells include their negatively charged surfaces, small size, and low specific gravity in addition to the dilute nature of the culture. Foam flotation, which is a subclass of Adsorptive Bubble Separation (ABS), can be used as an effective method for selective separation of contaminant from microalgae cultures as well as harvesting and enriching microalgae biomass due to its simplicity, and low capital & operating costs. Hydrothermal liquefaction (HTL) and in-situ transesterification are also potential conversion routes for biomass with high water content into bio-oil and biodiesel respectively.

The aim of the PI group is to use foam flotation to separate biological contaminants from microalgae culture, harvest and enrich microalgae continuously, convert the harvested biomass directly to biofuels, and identify potential intensification routes.

The main aim of the PI group in this work is to use a foam flotation column to separate biological contaminants from microalgae culture, harvest and enrich microalgae continuously and then convert the harvested biomass directly to biofuels without any downstream dewatering or drying processes. This is important because nearly 80% of energy cost is associated with drying. Some approaches are also proposed in this work to intensify the process through enhancing microalgae adsorption and reducing water flux of the rising foam. Additionally, the possibility of achieving oil extraction and eventual biodiesel production within the foam column is being assessed.

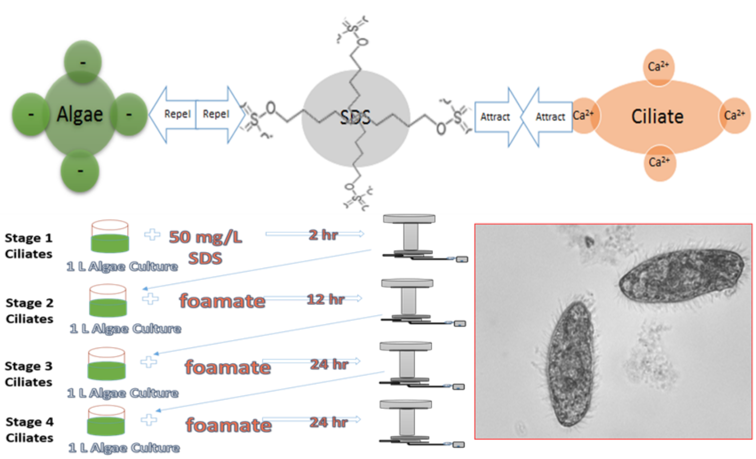

Fig 1. Top: hypothesized interaction between microalgae/SDS/ciliates. Bottom: Left: Experimental stages and procedure. Bottom Right: Ciliates T. *pyriformis*

Removal of ciliates contaminants from algae culture by foam column

Current approaches to microalgae culture management include growing species or strains that tolerate abiotic factors and chemicals such as pesticide and disinfectants. However, there remains the risk of causing inadvertent harm to the algae, or worse, to the culture personnel. Building upon work in surfactant-aided foam flotation, which demonstrated the efficacy and economic merits of the approach for dewatering and pre-processing microalgae biomass, the use of foam flotation for the removal of contaminating ciliate (*T. pyriformis*) from cultures of *Chlorella vulgaris* was investigated. Leveraging on the differences in microalgae surface charges (negatively charged) and ciliates (positively charged) we proposed a selective extraction of the ciliates from the contaminated culture using an anionic surfactant SDS (Fig 1) and the foam column. However, we could not achieve 100% ciliate removal from the culture, requiring high concentration of SDS (50 mg L^{-1}) to kill the ciliates and later recover the spent SDS for reuse.

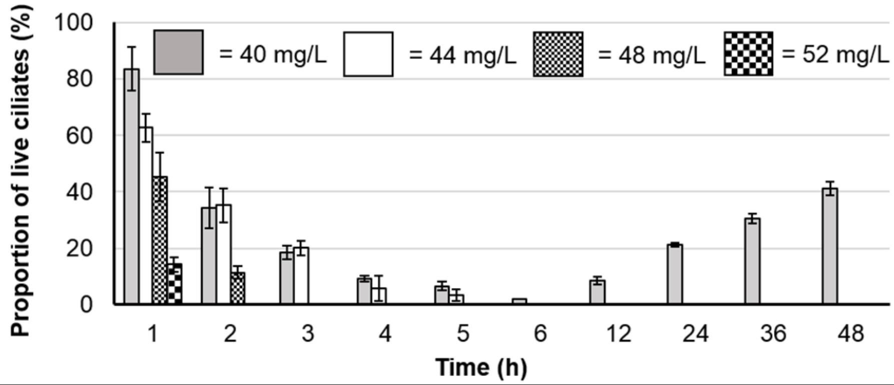

Fig 2. Survival and population regrowth of *T. pyriformis* following exposure to a range of SDS concentrations. All ciliates had died within six hours with the exception of the 40 mg/L treatment. Mean ± St. Dev.

The results demonstrated that high concentrations of SDS (>40 mg L^{-1}) are fatal for the ciliates as shown in Figure 2. Based on this knowledge of the susceptibility of *T. pyriformis* to SDS, a 50 mg L^{-1} concentration was used to treat four one litre batch algae cultures. Following treatment, the algae cultures grew ciliates free, increasing to 6.28×10^8 from 1.44×10^8 cells mL^{-1} within seven days. The microalgae were also found to be free of cell damage. Ciliates control using a multistage SDS recovery and reuse strategy proved to be faster (15 h 30 min) and offers an economic advantage over other ciliates chemical control measures.

Continuous harvesting of microalgae

Previous harvesting research by foam column has been performed in batch or semi-batch modes which are unsuitable for high throughput applications such as the production of biofuels. Furthermore, commercial scale algae production is usually done in continuous or semi-continuous modes. Continuous harvesting is therefore necessary for better process synergy. Continuous harvesting by foam flotation offers an additional advantage of controlling microalgae recovery or enrichment, or both. The foam flotation process involves generating bubbles by introducing air through a porous media. Destabilised microalgae and free surfactant will adsorb onto bubbles and are removed from the column in the form of foam as shown in Fig 3.

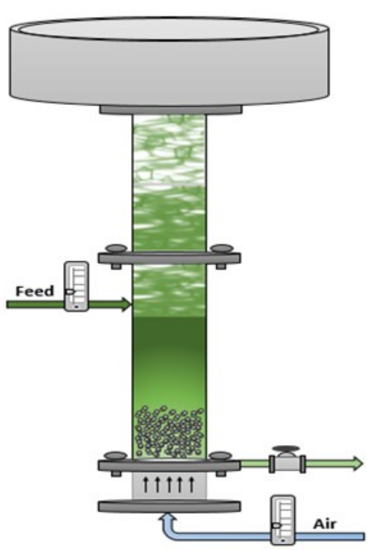

Fig 3. Schematic diagram of the continuous foam flotation column

Different surfactant types (cationic, anionic, and non-ionic) have been used and the amount of surfactant adsorbed to microalgae has been measured. This is important for identifying the surfactant that can best enhance the weak hydrophobicity of microalgae and also determine the amount of un-adsorbed surfactant required to stabilise the foam. The effect of all process factors has also been investigated using full factorial and response surface designs. These factors were surfactant concentration, air flow rate, feed flow rate, column height, liquid pool depth, and bubble size. The trials were conducted on both freshwater and marine microalgae including *Chlorella vulgaris, Dunaliella Salina, Nannochloropsis Oculata, Isochrysis* *galbana,* and *Tetraselmis suecica*. The recovery efficiencies and their corresponding enrichments for three of the above species based on the optimised factors are shown in Fig 4. Continuous foam flotation column showed a very low power consumption of 0.052-0.056 KWh per 1000 L of algae culture, with a total suspended solid yield that compares favourably with other commonly used harvesting techniques (as shown in table 1).

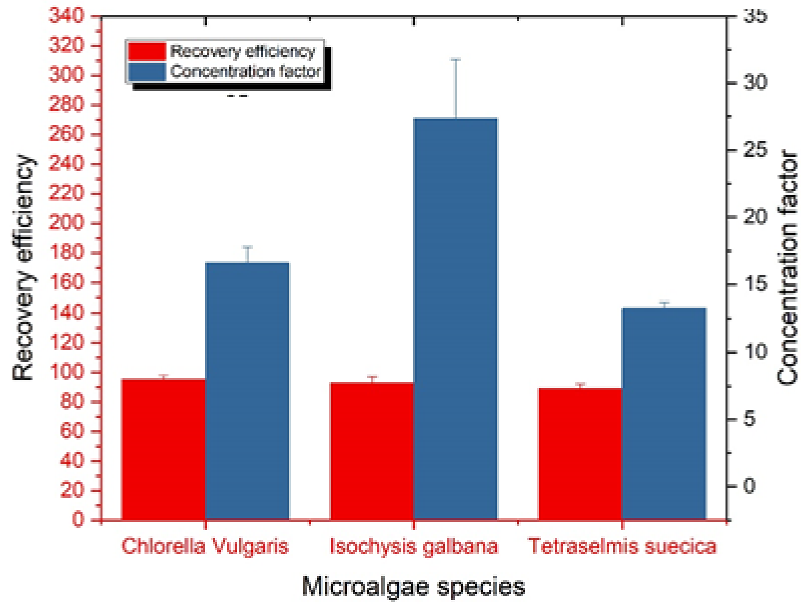

Fig 4. Recovery efficiency & concentration factor of three microalgae species

Table 1. Energy consumption, total suspended solids (TSS) and concentration factor (CF) of different microalgae harvesting techniques. Where reported, the recovery efficiency (RE%) is given in parentheses

Harvest Method	Operational Mode	Microalgae	Energy Consumption (KWh m^{-3})	TSS (%)	CF and RE
Dissolved air flotation	Batch	Multi-strain *Chlorella/ Scenedesmus*	7.6	5	N/A (85)
Electro-flotation	Batch	Multi-strain *Chlorella/ Coelastrum*	Very high, N/A	3—5	N/A
Foam flotation by Jameson cell	N/A	*Tetraselmis* sp. *M8*	N/A	N/A	23 (99)
Foam flotation (this study) based on optimised factors	Continuous	*Chlorella vulgaris*	0.052—0.056	2.7—5.6	173—415 (70—95)

Effect of methanol on microalgae separation process using foam flotation

This work investigated the feasibility of oil extraction and algae (*Chlorella vulgaris*) separation in a foam column by either adding methanol to the top of the foam column or mixing it with the algae culture in the base of the foam column. The ratios of the volume of methanol:volume of algae culture were in the range 10:90–80:20 and cationic surfactant (CTAB) concentrations were in the range 30–350mg/L. When methanol was added from the top of the column a concentration of 200mg/L CTAB was needed to achieve a stable foam. Comparatively, only 30mg/L CTAB was enough to generate a stable foam when methanol was mixed with the algae culture fed into the base of the column. Comparing the two scenarios in terms of the amounts of methanol and CTAB required, feeding methanol to the top of the foam column required 20ml methanol for 500ml of culture but higher CTAB (200mg/L). Mixing 500 ml methanol with 500 ml of culture in the base of the foam column only required 30mg/L of CTAB. Reducing the amount of methanol fed to the base of the foam column would allow for even less CTAB. By feeding methanol to the top of the foam column an algae concentration factor of 5.6 was achieved. When 10% methanol by volume of culture was fed to the base of the column the algae concentration factor was 142 at the rate of 70mL/min of feed and 1L/min air flow. It was found that algae separation is possible with the foam column in the presence of methanol as long as methanol does not make up more than 50% by volume of the liquid in the base of the column.

The material costs of the foam column were compared with another combined harvesting and extraction process for algae using Nano-clay. The foam column achieved a concentration factor of 142 at cost of at £2.10 per litre of culture whereas the Nano-clay process achieved a concentration factor of 5.9 at cost of about £39.60 per litre of culture medium.

Table 2. Summary of column experiments; A, B, and B* respectively represent scenarios where: (i) methanol was added from top, (ii) methanol was mixed with solution before adding surfactant, and (ii) methanol was added to already mixed algae culture with surfactant. Yes/No indicate whether the algae separation was achieved.

% Meth	30 mg/L (CTAB)	40 mg/L (CTAB)	50 mg/L (CTAB)	70 mg/L (CTAB)	80mg/L (CTAB)	100 mg/L (CTAB)	150 mg/L (CTAB)	200 mg/L (CTAB)	>350 mg/L (CTAB)
25A	No	No	No	No	No	No	No	Yes	Yes
25B	No	No	No	Yes	Yes	Yes	Yes	Yes	Yes
25B*	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes
50A	No	No	No	No	No	No	No	Yes	Yes
50B	No	No	No	No	No	Yes	Yes	Yes	Yes
50B*	No	No	No	Yes	Yes	Yes	Yes	Yes	Yes
75A	No	No	No	No	No	No	No	No	No
75B	No	No	No	No	No	No	No	No	No
75B*	No	No	No	No	No	No	No	No	No