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(54) Title: FLOW SYNTHESIS OF RDX

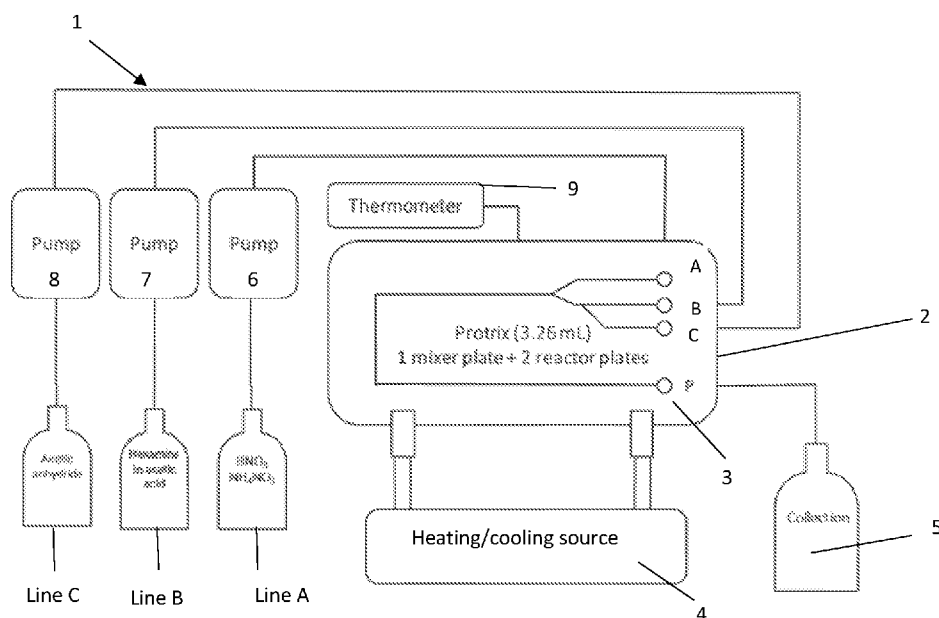


Fig 1

(57) Abstract: The invention relates to a method for the flow synthesis manufacture of RDX, comprising the steps of i. preparing input flow reagent A comprising greater than 95% concentration nitric acid and NH_4NO_3 ii. preparing input flow reagent B comprising hexamine dissolved in acetic acid; iii. preparing input flow reagent C, comprising acetic anhydride; iv. causing the input flow reagents A, B and C to enter a flow reactor, v. maintaining the reaction chamber to less than 90°C , vi. causing the input flow reagents to react in the reactor.

KH, KN, KP, KR, KW, KZ, LA, LC, LK, LR, LS, LU, LY,
MA, MD, MG, MK, MN, MU, MW, MX, MY, MZ, NA,
NG, NI, NO, NZ, OM, PA, PE, PG, PH, PL, PT, QA, RO,
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FLOW SYNTHESIS OF RDX

The following invention relates to methods of producing explosives from the indirect nitration of hexamine by flow synthesis. Particularly to a method of producing RDX.

5 Before the present invention is described in further detail, it is to be understood that the invention is not limited to the particular embodiments described, as such may, of course, vary. It is also to be understood that the terminology used herein is for the purpose of describing particular embodiments only, and is not intended to be limiting, since the scope of the present invention
10 will be limited only by the appended claims.

According to a first aspect of the invention there is provided a method for the flow synthesis manufacture of RDX, comprising the steps of

- i. preparing input flow reagent A comprising greater than 95% concentration nitric acid and NH_4NO_3 ;
- 15 ii. preparing input flow reagent B comprising hexamine dissolved in acetic acid;
- iii. preparing input flow reagent C, comprising acetic anhydride;
- iv. causing the input flow reagents A, B and C to enter a flow reactor,
- v. maintaining the flow reactor to less than 90 °C,
- 20 vi. causing the input flow reagents to react in the reactor.

The use of flow synthesis, (ie use of a pumped micro reactor), provides a facile means of preparing RDX at both laboratory R&D scale of ~100 g/hr and a larger industrial scale at multiple Kg/hr. The ability to add further flow reactors in
25 parallel allows the ability to scale-out, rather than traditional scale-up production, with the associated dangers of forming +100 Kgs of RDX explosive in a single reactor vessel. Further, it also reduces the volume of highly concentrated acid, at any given time, when compared to a large reactor vessel as found in batch process. The use of flow synthesis allows for the continuous removal and safe
30 stowage of final explosive product RDX material from the flow reactor or flow

reactors, to avoid the build-up of large quantities of explosive material. This may allow explosive processing buildings to process a greater mass of explosive per unit of time and/or associated safety distances to be reduced, as the explosive material may be distributed to safe areas, away from the flow reactor, as it is
5 synthesised.

In step i, the input flow reagent A may preferably be 99% concentration nitric acid. The ammonium nitrate may be present in the nitric acid in the range of from 50%wt to 75.5%wt.

In step ii, the flow reagent B, the hexamine may be dissolved in acetic acid
10 in the range of from 5%wt to 37.8%wt, preferably, in the range of from 30 %wt to 37.8%wt, preferably a saturated solution. Preferably the acetic acid is glacial acetic acid (anhydrous acetic acid).

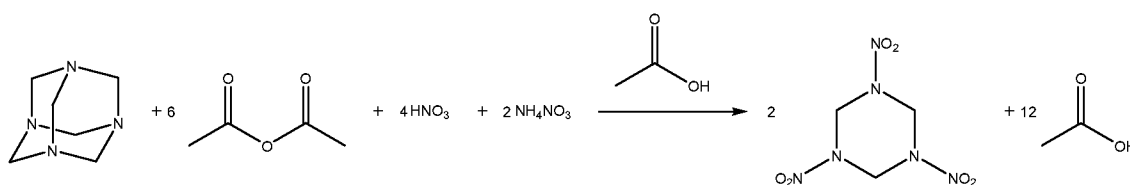
In step iii, preparing input flow reagent C, the acetic anhydride may be anhydrous ie dried, input flow reagent C may optionally comprise glacial acetic
15 acid.

In step iv, the input flow reagents A, B and C are pumped and combined in the flow reactor, a micro flow reactor, such that there is a continuous flow of the materials, and that the reaction occurs within a chamber/cell in the flow reactor. Preferably the total nitric acid concentration in the flow reactor may be in
20 the range of from 90- 99%.

There is a balance of reaction time vs maximum theoretical yield, ie time to pass the reactants through the flow reactor, and waiting for the maximum yield. In one arrangement the input flow reagents may be caused to be passed through the reactor for less than 45 seconds, preferably less than 15 seconds. This allows
25 for a fast throughput of reagents, to provide a high throughput, rather than the maximum theoretical yield.

In one arrangement the input flow reagent B and input flow reagent C may be premixed together, before being reacted with flow reagent A. The first step of the synthesis reaction is the reaction between the hexamine and the acetic
30 anhydride.

In a further arrangement, input flow reagent B and input flow reagent C may be passed through a first flow reactor, the output of the reaction product of input flow reagent B and input flow reagent C may then be passed through a second flow reactor, such that said reaction product of input flow reagent B and input flow reagent C, being reacted with flow reagent A to form RDX.



In a preferred arrangement the molar ratio of nitric acid:hexamine in the reactor is at least 3:1, this is below the stoichiometry of the reaction, it will provide a yield, but will furnish a sub optimal yield, preferably, the ratio of nitric acid:hexamine may be greater than 4:1, preferably 6:1, more preferably in the range of from 9:1 to 30:1. It is desirable to reduce the amount of nitric acid, to avoid wasting material and to avoid recycling any excess nitric acid, however, at ratios below 3:1, the reduced acid levels can lead to reduced yields and crystallisation, ie precipitation of the products in the flow reactor.

In a preferred arrangement the molar ratio of ammonium nitrate:hexamine in the flow reactor in step iv, is in the range of from 1:1 to 10:1.

The reaction once it has commenced is exothermic, therefore the temperature has to be controlled ie maintained, to ensure that the reaction does not cause too much heat to be generated. The temperature of the flow reactor may be maintained in the range of from 20 °C to 90 °C, preferably 50 °C to 90 °C,, more preferably in the range of 75°C to 90°C. At temperatures in excess of 90 °C there may be formation of acetyl nitrate, a highly undesirable side product.

The reactor may be subjected to both heating and cooling to retain the temperature within the working range. The flow reactor may preferably be formed from a ceramic, such as for example a non-oxide engineering ceramic, such as silicon carbide, which provides high thermal conductivity, good chemical compatibility and a high surface area, which allows precise thermal control of the

reaction. The non-oxide ceramics have a thermal conductivity orders of magnitude larger than borosilicate glass.

The flow reactor may be heated and cooled by any means, preferably the flow reactor is a liquid cooled/heated reactor, rather than air cooled. The reactor preferably has an integrated heat exchanger, and the use of a liquid as the working fluid, provides increased thermal control of the reaction. The temperature may be monitored by any measurement means, the flow reactor may be heated and/ or cooled by any suitable means such as for example water circulators or alternatively electric heater/coolers. The flow reactor may have different heating/cooling temperature controlled zones.

The flow reactor may comprise a mixing zone, comprising at least one mixing plate to mix together the input flow reagents A, B and C, said mixing zone maintained at a first temperature in a first temperature zone, and a reaction zone, comprising at least one residence plate which forms the microchannel path in which the reaction occurs, said reaction zone maintained at a second temperature in the second temperature zone.

The mixing zone is where the input flow reagents are brought together to mix in order to start a chemical reaction. A flow reactor may comprise more than one mixing zone, as further input flow reagents may be added after a first reaction has completed.

The mixing zone may comprise at least one further residence plate, such that the mixing plate and the further residence plate are maintained at the first temperature zone. The mixing of chemicals, and typically the case for the synthesis of energetic materials, is an exothermic reaction. Whilst the mixing plate may experience an exothermic reaction, the initial transfer into a further residence plate (i.e. the core of the flow reactor where the reaction takes place), the initial reaction may still be highly exothermic and the removal of excess heat, may help to control the reaction, especially from a safety perspective.

The reaction zone may comprise one, two or a plurality of residence plates, all maintained at the second temperature zone. There may be a third, fourth or further temperature zones, within the reactor- each temperature zone being held at a third, fourth or further temperature.

5 The first temperature zone may comprise a first water/oil circulator and the second temperature zone may comprise a second water/oil circulator.

The product may be isolated, such that at step vii, causing the output flow to be quenched, after the reagents have passed through the reactor. A quenching agent may be is an aqueous solution, such as to cause precipitation of RDX.

10

It may be undesirable to rapidly cool the mixture, as this reaction furnishes unwanted side reaction products that are only sparingly soluble.

The actual flow rate of input flow reagent A may be μL through to millilitres
15 to litres, depending on the capacity of the flow cell.

The output mixed flow is quenched, to stop the reaction and to cause precipitation of the RDX product. The output flow may be transferred in to a large volume of quench medium or mixed in a mixing chamber or preferably passed to
20 a flow crystallisation apparatus.

Experimental reagents

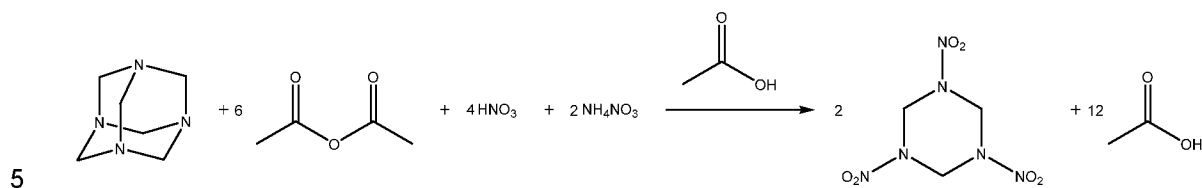
99 % HNO_3 was purchased from Honeywell in a 500 mL quantity. Cat. 84392-500ML, Lot. No. I345S.

25 70 % HNO_3 was purchased from Fisher scientific in a 2.5 L quantity. Code: N/2300/PB17. Lot: 1716505.

Hexamine was purchased from Sigma-Aldrich in a 250 g quantity. Cat. 797979-250G, Lot. No. MKCJ7669.

Flow reactor used: Protix

Experimental



A solution of ammonium nitrate (AN) dissolved in nitric acid was prepared by the addition of ammonium nitrate (45 g) to nitric acid (99%, 138.75 g) whilst stirring, once the ammonium nitrate had fully dissolved the solution was then attached to Line A of the Protix reactor. A second solution consisting of hexamine (hex) dissolved in acetic acid was prepared by the addition of hexamine (31.5 g) to glacial acetic acid (50 g) whilst stirring followed by sonication ensure full dissolution of the hexamine. This was then attached to Line B of the Protix reactor. Acetic anhydride was attached to Line C of the Protix reactor. Each line included a Vapourtech SF-10 pump and thermal control was provided by a Julabo heating circulator, which heated the reactor to 75 °C.

The ammonium nitrate(AN)/HNO₃ solution attached to line A was introduced to the reactor first at a rate of 10 mL/min, once nitric acid solution was observed exiting the product port, lines B and C were started at flow rates of 2.5 and 5.25 mL/min respectively. After equilibration, the product was collected as a clear yellow solution directly into an empty collection vessel for 2 minutes (Exp F003). The flow rate of Line A was reduced in subsequent experiments (Exp F004-008) with Lines B and C kept constant. Details of the reaction condition are shown in Table 1.

A clear yellow solution was observed in the collection vessels for the experiments with flow rates of 10-8 mL/min. A precipitate was observed in the collection vessels for the experiments with flow rates of 7-5.3 mL/min.

	Reagents	Flow Rate (mL/min)
Solution B	Hexamine/AcOH	2.5
Solution A	AN/HNO3	Variable
Solution C	Ac2O	5.24

Molar Ratios to Hexamine	
AcOH:Hexamine	3.71
Ac2O:Hexamine	6.99

The solution B (hexamine and acetic acid (AcOH) and solution C (acetic anhydride Ac₂O) were fixed, to allow the study of the variable of solution A (ammonium nitrate(AN) and nitric acid (HNO₃)).

5

Exp	Line A (mL/min)	Line B (mL/min)	Line C (mL/min)	T (°C)	Collection Time (minutes)	Residence time (s)
F003	10	2.5	5.24	75	2	11.0
F004	9	2.5	5.24	75	2	11.7
F005	8	2.5	5.24	75	2	12.4
F006	7	2.5	5.24	75	2	13.3
F007	6	2.5	5.24	75	2	14.2
F008	5.3	2.5	5.24	75	2	15.0

Table 1. List of reaction conditions for experiments F003-008.

All six of the reactions were diluted with water and then heated at ~95 °C for 30 minutes to remove any linear nitramines that may have formed. Once cooled to room temperature, the solids present in each reaction were filtered and washed with water.

10

Molar ratios, yields and percentages of RDX and HMX have been calculated and are shown in Table 2. The relative percentages of product was determined using their relative integrals from the ¹H NMR (500 MHz, CDCl₃) spectrum of the product mixture.

15

Exp	Line A Flow Rate (mL/min)	AN:Hexamine	HNO ₃ :Hexamine	Yield	% RDX	% HMX
F003	10	6.21:1	24.34:1	21.40%	98.97%	1.03%
F004	9	5.59:1	21.91:1	22.70%	98.54%	1.46%
F005	8	4.97:1	19.47:1	24.80%	97.45%	2.55%
F006	7	4.35:1	17.04:1	24.50%	96.70%	3.30%
F007	6	3.73:1	14.6:1	25.40%	95.69%	4.31%
F008	5.3	3.29:1	12.9:1	28.60%	94.50%	5.50%

Table 2. List of molar ratios, yields and percentages of RDX and HMX for experiments F003-008.

There are three variables that change due to the flow rate here, residence time, acid concentration and ammonium nitrate concentration. Increasing the residence time may have a positive effect on yield, but this needs to be balanced with throughput. The HMX is a side product in this reaction, and is itself a useful product.

This synthetic route allows for a rapid transit time of reagents through the reactor, with minimal impurities.

Whilst the invention has been described above, it extends to any inventive combination of the features set out above, or in the following description, drawings or claims.

Exemplary embodiments of the device in accordance with the invention will now be described with reference to the accompanying drawings in which:-

Figure 1 show a flow reactor arrangement.

Figure 2 shows a two stage flow reactor arrangement.

Figures 3 A to E shows alternative two stage flow reactor arrangements.

Turning to figure 1 there is provided a schematic of a flow reactor 1 (a micro reactor) The reagents in Line A, Line B and Line C, are fed via associated pumps 6, 7, 8, to the Protrix flow reactor 2, in the arrangement shown, lines B and C briefly premix before being mixed with line A, however the reactor cell can be configured to allow all three Line A, B and C to enter at the same time. A thermometer 9 monitors the temperature in the flow reactor 2, and applies heating/cooling from the heating and cooling source, which may be water jackets or electrical heating and cooling elements. The reactants flow through the reactor plate 2, and emerge at the product exit 3, which feeds into a collection vessel 5, for further processing.

Turning to figure 2 there is provided a schematic of a flow reactor 10 for the Bachmann reaction. The reagents in Line A, and Line B and Line C are fed via associated pumps 16, 17 and 18, to the Protrix flow reactor 12. The reactor cell 12 in this arrangement has been configured to allow all three Line A, B and C to enter at the same time. A thermometer 19a monitors the temperature in the

mixing zone 20 of the flow reactor 12, and controls the temperature by means of the heating and cooling source 14a which is water circulator (T1). The reactants flow through the residence plate in the reaction zone 21, in the reactor cell 12, where thermometer 19b monitors the temperature in the reaction zone 11, and
5 controls the temperature by means of heating or cooling from the heating and cooling source 14b, a water circulator (T2).

The final reactant emerges at the product exit 13, which feeds into a collection vessel 15, for further processing.

Turning to figure 3 A-E shows different arrangements of the reactor cell.
10 In arrangement A, there is a mixing zone 31 with a mixing plate which has its own water circulator T1 to control the temperature.

The reaction zone 30 comprises three residence plates 1, 2, 3, which have their own water circulator T2 to control the temperature. A service plate provides a thermal barrier between the mixing zone 31 and the reaction zone 30, to ensure
15 there are discreet temperature zones. Further service plates and further water circulators may be used to create further temperature zones.

Arrangements B and C, show further residence plates being sequentially added to the mixing zone and removed from the reaction zones. Alternatively more residence plates could be added to both the mixing zone and reaction
20 zones.

Arrangements D and E show the removal of residence plates, which reduces the residence time of the reagents in the reactor. The output from one reactor cell comprising the mixing/reaction zone, could be fed into a second or third reactors where further reagents are needed to accomplish multi step
25 reaction pathways.

Claims

1. A method for the flow synthesis manufacture of RDX, comprising the steps of
 - 5 i. preparing input flow reagent A comprising greater than 95 % concentration nitric acid and NH_4NO_3
 - ii. preparing input flow reagent B comprising hexamine dissolved in acetic acid;
 - 10 iii. preparing input flow reagent C, comprising acetic anhydride; optionally acetic acid;
 - iv. causing the input flow reagents A, B and C to enter a flow reactor;
 - v. maintaining the flow reactor to less than 90°C ;
 - vi. causing the input flow reagents to react in the reactor.
- 15 2. The method according to claim 1, wherein in step i the input flow reagent A is 99% concentration nitric acid.
3. The method according to any one of the preceding claims wherein the ammonium nitrate is present in the nitric acid in the range of from 50%wt to 75.5%wt.
- 20 4. The method according to any one of the preceding claims, wherein step ii the hexamine is dissolved in acetic acid in the range of from 5%wt to 37.8%wt
5. The method according to claim 4, wherein the hexamine is dissolved in acetic acid in the range of from 30%wt to 37.8%wt.
6. The method according to any one of the preceding claims, wherein in step
25 iv, the total nitric acid concentration is in the range of from 90-99%, in said flow reactor.
7. The method according to any one of the preceding claims, wherein input flow reagent B and input flow reagent C are premixed before being reacted with flow reagent A.

8. The method according to any one of the preceding claims, wherein the molar ratio of nitric acid:hexamine in the reactor is at least 3:1.
9. The method according to claim 8, wherein the molar ratio of nitric acid:hexamine is in the range of from 9:1 to 30:1.
- 5 10. The method according to any one of the preceding claims, wherein the molar ratio of ammonium nitrate:hexamine in the reactor is in the range of from 1:1 to 10:1.
11. The method according to any one of the preceding claims causing the input flow reagents to react in the reactor for less than 45 seconds.
- 10 12. The method according to any one of the preceding claims, wherein the temperature of the reactor is in the range of 20 °C to 90 °C.
13. The method according to any one of the preceding claims, wherein the material of the flow reactor cell comprises a non-oxide engineering ceramic.
14. The method according any one of the preceding claims, wherein the
15 reactor comprises
- a mixing zone, comprising at least one mixing plate to mix together the input flow reagents A, B and C, said mixing zone maintained at a first temperature in a first temperature zone, and
- a reaction zone, comprising at least one residence plate which forms the
20 microchannel path in which the reaction occurs, said reaction zone maintained at a second temperature in the second temperature zone.
15. A method according to claim 14,
- i. preparing input flow reagent A, comprising hexamine pre-dissolved in water or glacial acetic acid,
- 25 ii. preparing input flow reagent B comprising greater than 95% concentration nitric acid,
- iii. causing the input flow reagents A and B to enter the mixing zone at a flow rate, so as to cause a total nitric acid concentration of greater than 90%, in said flow reactor,

- iv. cooling the mixing zone to less than 15°C,
 - v. causing the mixed flow to pass through into the reaction zone, heating the reaction to greater than 25°C
 - vi. optionally quenching the output flow from the reaction zone, to cause precipitation of RDX.
- 5

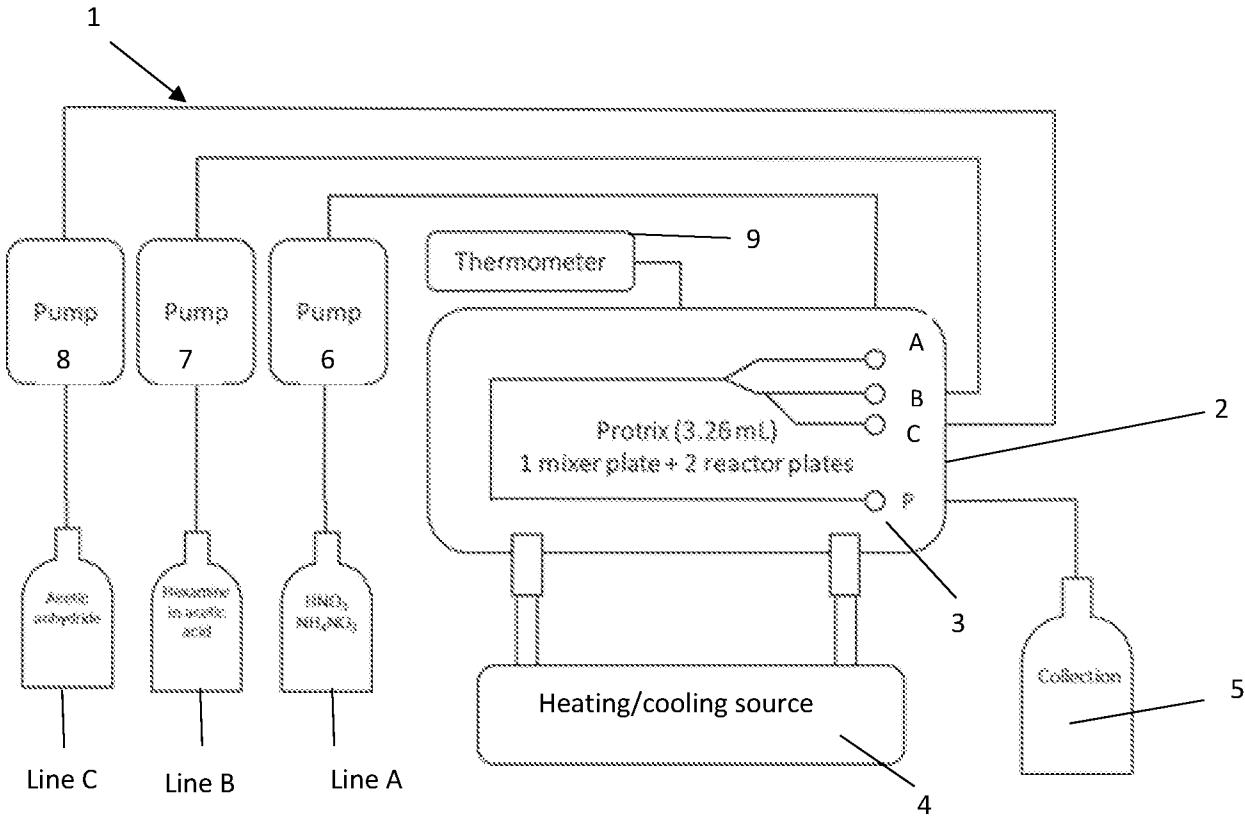


Fig 1

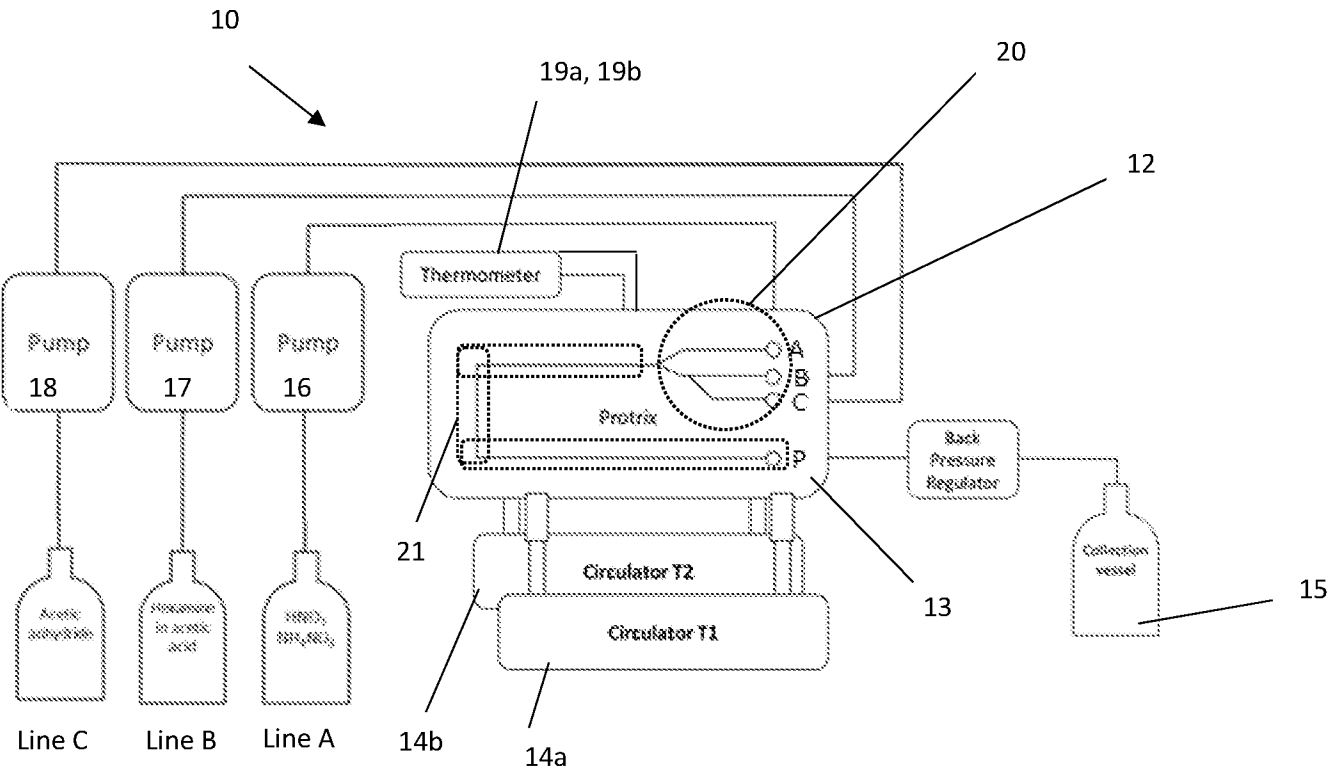


Fig 2

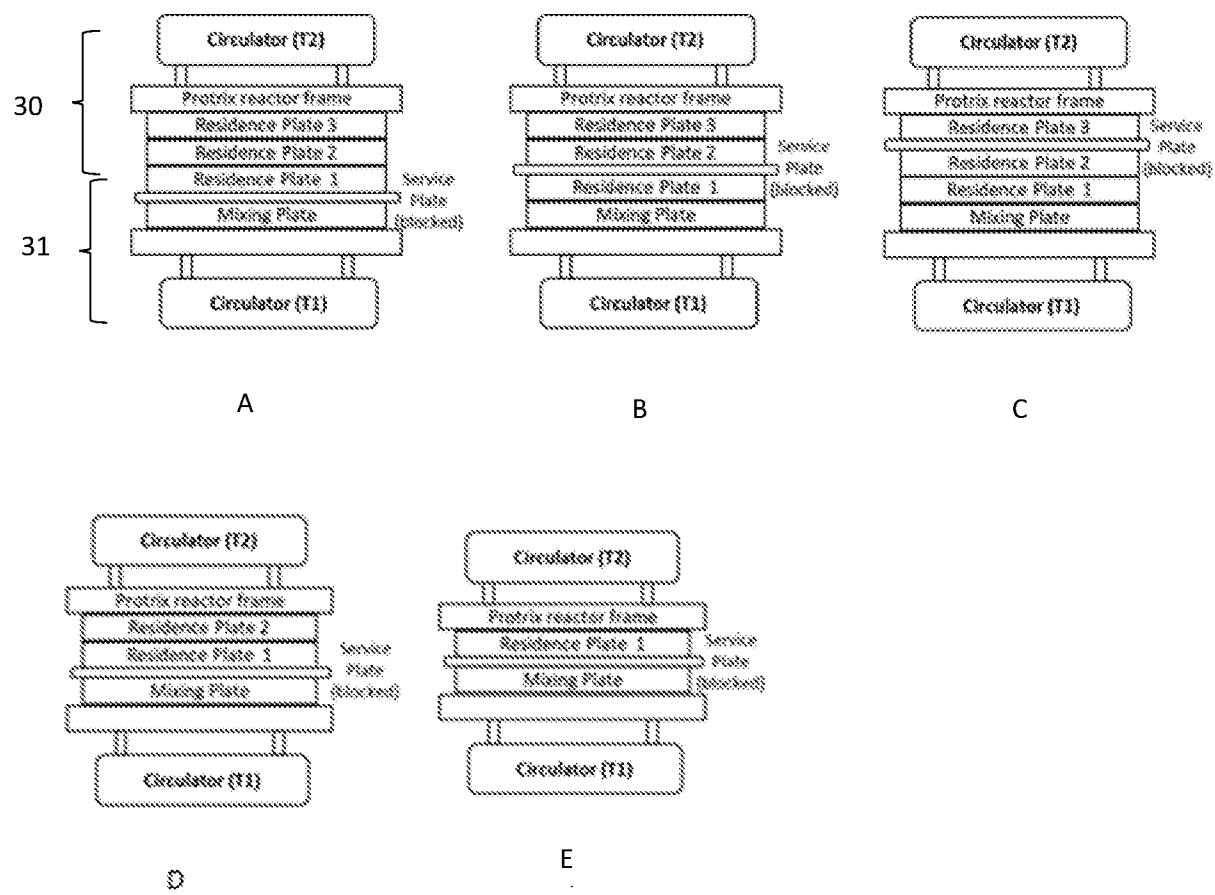


Fig 3

INTERNATIONAL SEARCH REPORT

International application No
PCT/GB2024/053064

A. CLASSIFICATION OF SUBJECT MATTER INV. C06B25/34 C07D251/06 ADD.		
According to International Patent Classification (IPC) or to both national classification and IPC		
B. FIELDS SEARCHED Minimum documentation searched (classification system followed by classification symbols) C06B C07D Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched Electronic data base consulted during the international search (name of data base and, where practicable, search terms used) EPO-Internal		
C. DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	CA 702 021 A (US GOV SEC ARMY) 19 January 1965 (1965-01-19) figure 1; example 1 -----	1 - 15
E	WO 2025/037081 A1 (BAE SYSTEMS PLC [GB]) 20 February 2025 (2025-02-20) page 16, line 24 - page 17, line 8 -----	1, 14
A	WO 2022/123216 A1 (BAE SYSTEMS PLC [GB]) 16 June 2022 (2022-06-16) claim 1 ----- - / - -	1 - 15
☒ Further documents are listed in the continuation of Box C. ☒ See patent family annex.		
* Special categories of cited documents : "A" document defining the general state of the art which is not considered to be of particular relevance "E" earlier application or patent but published on or after the international filing date "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified) "O" document referring to an oral disclosure, use, exhibition or other means "P" document published prior to the international filing date but later than the priority date claimed "T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention "X" document of particular relevance;; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone "Y" document of particular relevance;; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art "&" document member of the same patent family		
Date of the actual completion of the international search 26 February 2025		Date of mailing of the international search report 11/03/2025
Name and mailing address of the ISA/ European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Fax: (+31-70) 340-3016		Authorized officer Kappen, Sascha

INTERNATIONAL SEARCH REPORT

International application No

PCT/GB2024/053064

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	JADHAV PANDURANG M; PRASANTH HIMA; PANDEY RAJ K; SHINDE UJWALA R; DESAI SHASHIKANT A: "Optimization and Kinetics Evaluation of Bachmann Process for RDX Synthesis", INTERNATIONAL JOURNAL OF CHEMICAL REACTOR ENGINEERING, 20170907 BERKELEY ELECTRONIC PRESS, BERKELEY, CA, US, vol. 16, no. 4, 7 September 2017 (2017-09-07), pages 20170061-1, XP055894529, ISSN: 1542-6580, DOI: 10.1515/ijcre-2017-0061 Retrieved from the Internet: URL: http://dx.doi.org/10.1515/ijcre-2017-0061 pages 2-3: experimental details -----	1 - 15

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No
PCT/GB2024/053064

Patent document cited in search report		Publication date	Patent family member(s)	Publication date
CA 702021	A	19-01-1965	NONE	

WO 2025037081	A1	20-02-2025	WO 2025037081 A1	20-02-2025
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