EUROPEAN ROADMAP OF PROCESS INTENSIFICATION

- TECHNOLOGY REPORT -

TECHNOLOGY: **HEAT-INTEGRATED DISTILLATION**

TECHNOLOGY CODE: 2.1.4

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1. Technology

1.1 Description of technology / working principle

(Feel free to modify/extend the short technology description below)

Dividing wall columns represent an improved type of distillation column. They are equipped with one or more vertical partitions inside the column shell. In contrast to conventional distillation columns they are able to deliver pure side fractions. This feature reduces the number of necessary distillation columns in a separation sequence.

Energy savings in the range of 30 % can be obtained. Investment costs can be reduced to the same extent, as several separation steps can be integrated into a column shell and the number of heat exchangers is decreased. Additional advantages are improved yields and purities of the products in the case of thermally sensitive products. The range of applications of this distillation technology is broad. It covers different types of column internals. Various products – even with extreme purity demands – can be produced.

Compared to conventional distillation sequences (direct or inverse) dividing wall columns provide separation sequences which minimize the formation of additional entropy of mixing on the feed plate. This is the thermodynamic reason for the lower energy consumption.

As an alternative, dividing wall columns can be replaced by the arrangement of thermally coupled distillation columns. They provide the same energy savings, but lead to increased investment. Application of thermally coupled columns is indicated in cases with different pressures in the columns.

Retrofit of existing conventional columns can be made for dividing wall columns as well as for thermally coupled columns. Increased capacities can be expected in retrofit situations.

1.2 Types and "versions"

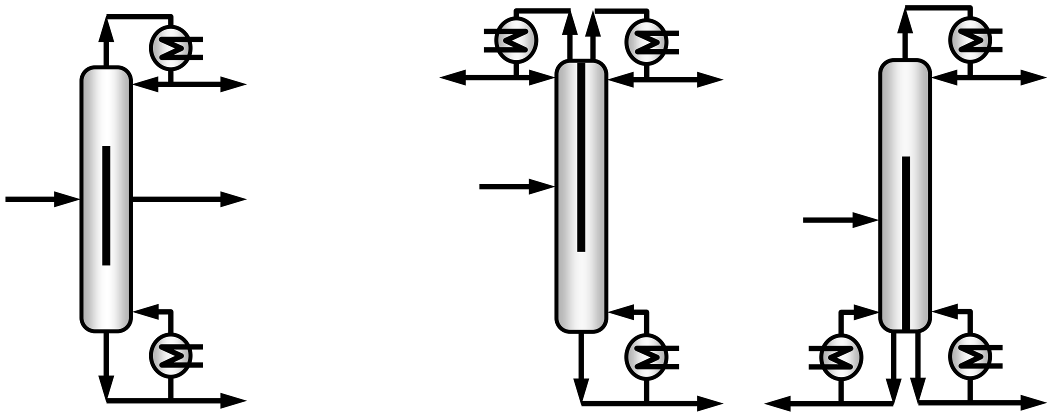
(Describe the most important forms/versions of technology under consideration, including their characteristic features, differences and similarities)

The standard configuration has a dividing wall in the middle section of the column. This type represents the thermodynamically optimal configuration which can avoid the formation of additional entropy of mixing on the feed plate. It offers lower investment costs and lower energy consumption.

The alternative column configuration has dividing walls fixed to the upper or the lower end of the column. This type represents merely an apparative integration of a side column to the main column, a side rectifier or a side stripper. Lower investment costs can be expected, but no energy savings.

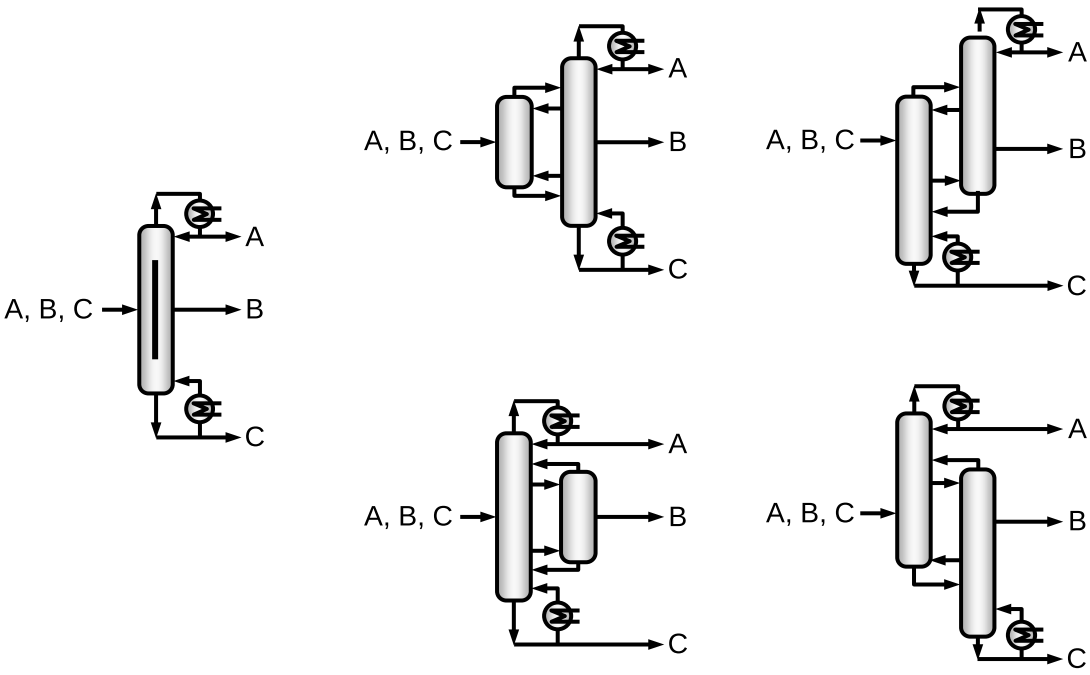
Dividing wall columns with the dividing wall in the middle section of the column have found broader application. They offer excellent chances for process integration.

Thermally coupled distillation columns represent an apparative alternative to dividing wall columns. They offer the same potential of energy savings. Investment costs are higher compared to dividing wall columns. They are primarily used in retrofit situations or in cases where different pressures in the columns are necessary.



Thermodynamically optimal
Lower investment
Lower energy consumption

Apparative integration
Lower investment



Thermally coupled columns as an alternative to a dividing wall column

1.3 Potency for Process Intensification: possible benefits

(In Table 1 describe the most important documented and expected benefits offered by the technology under consideration, focusing primarily on energy; CO_2 emission and costs, providing quantitative data, wherever possible. Add other benefits, if needed).

Table 1: Documented and expected benefits resulting from technology application

Benefit	Magnitude	Remarks
Energy savings	30 % on an average	Savings up to 45 % in production plants have been obtained

Investment savings	30 % on an average	Reduced number of column shells and heat exchangers
Reduced plot space	30 – 40 %	Reduced apparative equipment
Reduced thermal stress, better product qualities		Important in cases of thermally sensitive products
Reduced thermal stress, higher distillation yields		Important in cases of thermally sensitive products

1.4 Stage of development

Distillation columns with one dividing wall in the middle section have found industrial application. Arrangements with one or two pure side streams are in use. To a smaller extend columns with dividing walls at the upper or the lower ends are applied.

Control concepts are still a topic in scientific publications.

Different constructional concepts have been developed, e.g. removable non-fixed dividing walls, sealing concepts, lateral offset of the dividing wall, different heights of feed plates and side draws, construction elements for regular packings and trays, thermal insulation of the dividing wall, mixing elements at the ends of the dividing wall, liquid and vapor distribution devices.

Simulation programs are available. Equation-based programs show better convergence characteristics compared to programs with sequential algorithms.

Laboratory or pilot plant scale equipment is available.

Process integration is up to now constrained to columns with one dividing wall. Applications of columns with more than one dividing wall have not yet been reported. Further development is indicated.

2. Applications

2.1 Existing technology (currently used)

(Describe technology (-ies) that are conventionally used to perform the same or similar operations as the PI-technology under consideration)

Conventional side draw distillation columns. Side draw columns without dividing walls cannot deliver pure side fractions. The side product is contaminated either by the low boiling product when the side draw is located in the rectifying section respectively by the high boiling product, when the side outlet is placed in the stripping section. As the medium boiling product is in many cases the main product, contaminations usually cannot be tolerated.

To avoid contaminations side products have to be replaced by top or bottom products. This increases the number of necessary distillation columns.

2.2 Known commercial applications

(Is the technology broadly applied on commercial scale? In which process industry sectors is the technology most often applied: large volume chemicals – specialty chemicals & pharma – consumer products – ingredients based on agro feedstocks? What is the estimated number of existing applications? In Table 2 provide the most prominent examples of realized applications and provide their short characteristics)

Table 2. Industrial-scale applications of the technology (existing and under realization)

Sector	**Company - Process/Product name/type**	**Short characteristic of application**	**Production capacity/ Plant size**	**Year of application**	**Reported effects**
Chemical company	BASF	Wide field of applications, 50 existing columns in production scale, 10 – 15 columns under construction or planned	No limitations with respect to capacity	First start-up in 1985	Energy savings, investment savings, reduced plot space, better product qualities, higher yields
Chemical companies	Sasol Sumitomo, Condea, Cognis, Bayer and others	Sasol: olefins chemicals	Sasol: up to 5.2 m x 107 m		Energy savings, investment savings, improved product purities
Refineries, chemical companies	Veba Ruhröl Chevron BP CEPSA ExxonMobil Aral Aromatics UOP and others	Pyrol. gasoline pyrol. gasoline aviation gasoline paraffins xylenes toluene hydrocarbons			Energy savings, investment savings, simple retrofits, extractive distillation

2.3 Known demonstration projects

(Are there any demonstration projects known related to the technology under consideration? In which process industry sectors are those projects carried out: large volume chemicals – specialty chemicals & pharma – consumer products – ingredients based on agro feedstocks? In Table 3 provide the short characteristics of those projects.)

Table 3. Demonstration projects related to the technology (existing and under realization)

Sector	**Who is carrying out the project**	**Short characteristic of application investigated, including product name/type**	**Aimed year of application**	**Reported effects**
Reactive distillation	EU project INSERT	Heterogeneous catalysis		Proof of concept reached

2.4 Potential applications discussed in literature

(Provide a short review, including, wherever possible, the types/examples of products that can be manufactured with this technology)

Reactive distillation is considered and has already been examined. No industrial applications up to now.

3. What are the development and application issues?

3.1 Technology development issues

(In Table 4 list and characterize the essential development issues, both technical and non-technical, of the technology under consideration. Pay also attention to "boundary" issues, such as instrumentation and control equipment, models, etc.) Also, provide your opinion on how and by whom these issues should be addressed)

Table 4. Technology development issues

Issue	Description	How and by whom should be addressed?
Reactive distillation	Vapor distribution at the lower end of the dividing wall, handling column internals with different pressure drops	Universities, apparatus manufacturers, chemical companies
Control	In-house information not available to the public	Chemical companies, universities
Cheap equipment	Further reduction of investment costs	Apparatus manufacturers
Multiple dividing walls	Improved process intensification	Apparatus manufacturers, chemical companies
Handling of high temperature differences across the column	Limitation of mechanical stress, appropriate construction concepts, inclination	Apparatus manufacturers, chemical companies

3.2 Challenges in developing processes based on the technology

(In Table 5 list and characterize the essential challenges, both technical and non-technical, in developing commercial processes based on the technology under consideration. Also, provide your opinion on how and by whom these challenges should be addressed)

Table 5. Challenges in developing processes based on the technology

Challenge	Description	How and by whom should the challenge be addressed?
Simulation	Simulation programs with good convergence characteristics are needed.	Software companies, universities
Experiments	Reliable apparative equipment needed.	Chemical companies, apparatus manufacturers, universities
Control concepts	Control systems for various situations should be established and checked by experience from	Chemical companies, Universities

	production scale columns. Dynamic simulations to handle short-term fluctuations of feed concentrations.	
Construction principles	Development of reliable equipment to obtain high purities.	Apparatus manufacturers, chemical companies

4. Where can information be found?

4.1 Key publications

(Provide the list of key publications in Table 6)

Table 6. Key publications on the technology

Publication	Publication type (research paper/review/book/ report)	Remarks
Kaibel, G., 1987, Distillation columns with vertical partitions, Chem. Eng. Technol. 10/1987, 92 - 98	Description of the technology	First paper in the field
Kaibel, G., Blass E., Köhler J., 1990, Thermodynamics – guideline for the development of distillation column arrangements, Gas Separation & Purification 1990 Vol. 4, 109 - 114	Research paper	Thermodynamic background, heat integration
Triantafyllou C., Smith R., 1992, The design and optimization of fully thermally coupled distillation columns, Trans IChemE, Vol. 70 Part A, March 1992, 118 - 132	Research paper	Design method
Wolff E., Skogestad S., 1995, Operation of integrated three-product (Petlyuk) distillation columns, Ind. Eng. Chem. Res., Vol. 34, No. 6, 1995, 2094 - 2103	Research paper	Control behaviour
Mutalib M., Smith R., 1998, Operation and control of dividing wall distillation columns, Part 1 : degrees of freedom and dynamic simulation, Trans IChemE, 3/1998, 76, part A, 308 - 318	Research paper	Control behaviour
Mutalib M, Smith R., 1998, Operation and control of dividing wall distillation columns, Part 2 : simulation and plant studies using temperature control, Trans IChemE, 3/1998, 76, part A, 319 – 334	Research paper	Control behaviour, experiments
Becker H., Godorr S., Kreis H., Vaughan J., 2001, Partitioned distillation columns – why, when & how, Chemical Engineering, January 2001, 68 – 74	Review	Case study
Halvorsen I., Skogestad S., 2003, Minimum energy consumption in multicomponent distillation. 1. V_{min} diagram for a two-product column, Ind. Eng. Chem. Res. 2003, 42, 596 – 604	Research paper	Design method
Halvorsen I., Skogestad S., 2003, Minimum energy consumption in multicomponent distillation. 2. Three-product Petlyuk arrangements, Ind. Eng. Chem. Res. 2003, 42, 605 – 615	Research paper	Design method
Halvorsen I., Skogestad S., 2003, Minimum energy consumption in multicomponent distillation. 3. More than three products and generalized Petlyuk arrangements, Ind. Eng. Chem. Res. 2003, 42, 616 – 629	Research paper	Advanced configurations, design method

Kaibel G., Miller C., Stroezel M., von Watzdorf R., Jansen H., 2004, Industrieller Einsatz von Trennwandkolonnen und thermisch gekoppelten Destillationskolonnen, Chem. Ing. Tech. 76, 3/2004, 258 - 263	Review	Industrial applica-tions, constructional details
Kaibel G., Miller C., Holtmann T., Schoenmakers H., 2005, Reaktivdestillation, Chem. Ing. Tech. 2005, 77 No. 11, 1749 - 1758	Review	Reactive distillation in dividing wall columns
Spencer, G., Plana Ruiz F., 2005, Consider dividing wall distillation to separate solvents, Hydrocarbon Processing, July 2005, 90 - 94	Hydrocarbons	Process revamp, lower capital and operating costs
Diehl T., Kolbe B., Gehrke H., 2006, Recovering pure aromatics, PQT Q2 2006, 127 - 131	Aromatics extractive distillation	Industrial appli-cation, savings
Parkinson G., 2007, Dividing wall columns find greater appeal, CEP, May 2007, 8 - 11	Review	Industrial applications
Müller I., Kenig E., 2007, Reactive distillation in a dividing wall column: rate-based modelling and simulation, Ind. Eng. Chem. Res., 2007, 46, 3709 - 3719	Research paper	Reactive distillation, simulations

4.2 Relevant patents and patent holders

(Provide the list of relevant patents in Table 7. Under "remarks" provide, where applicable, the names/types of products targeted by the given patent.)

Table 7. Relevant patents

Patent	**Patent holder**	**Remarks, including names/types of products targeted by the patent**
US 2 471 134: Fractionation apparatus	Standard Oil	1 side draw
EP 0 122 367: Destillationskolonne	BASF	2 side draws
DE 10 2004 004 672: Verfahren zur destillativen Gewinnung von reinen Isopropenylethern	BASF	2 side draws
DE 3302525: Destillationskolonne zur Zerlegung eines aus mehreren Frak-tionen bestehenden Zulaufproduktes	BASF	Multiple dividing walls, multiple side draws
EP 0141 356: Verfahren zur Gewin-nung eines konjugierten Diolefins und/oder Olefins aus einem C4- oder C5-Kohlenwasserstoffgemisch	BASF	Multiple dividing walls, extractive distillation
JP 2001 334 101: Distillation appara-tus and distillation method using the same	Kyowa Yuka	Multiple dividing walls
JP 2001 353 401: Distillation appa-ratus has partitions formed in dis-tillation regions obtained by par-titioning tower housing	Kyowa Yuka	Multiple dividing walls
DE 10 2005 002 129: Reaktor und Verfahren in einem Reaktor mit einem Reaktorinnenraum, der in zwei oder mehrere voneinander getrennte Reaktionsräume aufgeteilt ist	BASF	Multiple dividing walls, dividing walls in reactors
JP 2003 220 301: Distillation purification method has distillation column with four divided chambers to	JGC	Dividing walls at upper and lower ends of the column

separate components of different boiling points in raw material		
EP 0 126 288: Verfahren zur Durchführung von chemischen Reaktionen und gleichzeitiger destillativer Zerlegung eines Produktgemisches in mehrere Fraktionen mittels einer Destillationskolonne	BASF	Reactive distillation
DE 100 22 465 : Verfahren und Vorrichtung zur Aufarbeitung eines C4-Schnitts aus der Fraktionierung von Erdöl	BASF	Reactive distillation
DE 100 33 958: Verfahren zur Reaktivdestillation	BASF	Reactive distillation
DE 199 58 464 : Verfahren zur Extraktivdestillation und Kolonne zur Durchführung des Verfahrens	Krupp Uhde	Extractive distillation, integrated side column
EP 1 042 040: Rectifying column for extractive distillation of close-boiling or azeotropic mixtures	Krupp Uhde	Extractive distillation
WO 2001/ 79 389: Plant for the extraction of aromatic substances from a hydrocarbon mixture containing aromatic and non-aromatic substances	Krupp Uhde	Extractive distillation, aromatics
DE 100 19 196: Verfahren zur Gewinnung eines aus Benzol und Toluol oder Toluol und Xylol bestehenden Aromatenproduktes hoher Reinheit aus einem Nichtaromaten enthaltenden eng- oder azeotropsiedenden Vorprodukt und Anlage zur Druchführung des Verfahrens	Krupp Uhde	Extractive distillation BTX
WO 2002/45 811: Method and device for separating a substance mixture into the component parts thereof by means of extractive distillation in a separating wall column	Linde	Extractive distillation
DE 101 35 585: Verfahren und Vorrichtung zur Gewinnung einer reinen Benzol, Toluol und Xylole umfassenden Aromatenfraktion aus Raffinerieströmen durch Extraktivdestillation	Krupp Uhde	Extractive distillation, multiple dividing walls
EP 0 133 510: Verfahren zur Zerlegung eines azeotropen Stoffgemisches	BASF	Azeotropic distillation
DE 35 10 365: Verfahren zur destillativen Zerlegung eines Gemisches mittels einer Destillationskolonne	BASF	Separation of 2- or 3-component mixtures
EP 1 317 947: Improved batch distillation	Air Products and Chemicals	Discontinuous operation
US 2 295 256: Process and device for fractional distillation of liquid mixtures, more particularly petroleum	Brugma	Thermally coupled distillation columns
DE 35 14 365: Destillationssystem, bestehend aus zwei Destillationskolonnen zur destillativen energiegünstigen Zerlegung eines aus mehreren Fraktionen bestehenden Zulaufproduktes	BASF	Thermally coupled distillation columns
EP 0 380 001: Verfahren zur Abtrennung geringer Mengen einer	BASF	Thermally coupled distillation columns

Mittelsiederfraktion aus einem Flüssigkeitsgemisch		
EP 0 284 971: Verfahren zur Gewinnung von 1,3-Butadien	BASF	Thermally coupled distillation columns
DE 196 18 152: Verfahren zur Trennung von Rohester im DMT-Prozess	Hüls	Thermally coupled distillation columns
EP 1 080 766: Distillation of multicomponent fluid e.g. air, involves partial separation in first column, with mixture stream transferred one-way for final separation in second column	Air Products and Chemicals	Thermally coupled distillation columns
EP 0 640 367: Destillationskolonne zur Trennung eines Flüssigkeitsgemisches in mehrere reine Fraktionen	BASF	Thermal insulation of the dividing wall
US 6 645 350: Dividing wall column fractionation tray	UOP	Thermal insulation of the dividing wall
EP 1 378 282: Distillation apparatus	Sumitomo Heavy Industries	Lateral offset of the dividing wall
WO 1999/56 848 : Device and method for distillation	Sumitomo Heavy Industries	Lateral offset of the dividing wall
EP 1 127 601: Trennwandkolonne zur Auftrennung eines Vielstoffgemisches	BASF	Diagonal offset of the dividing wall
DE 101 63 335 : Trennwandkolonne mit ganz oder teilweise dampfförmigen Zulauf und/oder ganz oder teilweise dampfförmiger Seitenentnahme	BASF	Diagonal offset of the dividing wall
DE 43 36 983: Kolonne mit einer Trennwand	Montz	Construction details
DE 43 36 984: Kolonne für überhitzte Flüssigkeiten	Montz	Construction details, integrated flash-box
DE 43 36 985: Kolonne zum Durchführen thermischer Trennungen und/oder chemischer Reaktionen	Montz	Construction details, self-centering packing elements
DE 43 36 986: Kolonne mit Trennwand	Montz	Construction details
EP 0804 951: Trennwandkolonne zur kontinuierlichen destillativen Zerlegung von Mehrstoffgemischen	BASF	Unfixed wall elements
EP 1 088 577: Kolonnentrennwand	Montz	Unfixed wall elements
WO 2002/094 408: Fixing parts and seals for partition elements of a multi-section partition	Montz	Unfixed wall elements in trayed columns
EP 1 151 781: Trennwandkolonne	BASF	Unfixed wall
EP 0 684 060: Verfahren und Vorrichtung zur destillativen Trennung von Stoffgemischen	BASF	Adjusted liquid distribution
DE 103 52 294: Kolonne	Montz	Mixing elements at the ends of the dividing wall
EP 1 598 098: Trennwandkolonne	BASF	Construction for high temperature differences
EP 1 647 318: Dividing wall column	Air Products and Chemicals	Arrangement of regular packings in dividing wall columns
US 2 134 882: Fractionating apparatus and method of fractionation	Standard Oil	Dividing wall at lower end
US 2004/0182751: Low capital implementation of distributed distillation in ethylene recovery	BP America	Dividing wall at lower end, ethylene
EP 1 108 965 : Process for distillation of multicomponent fluid suitable for	Air Products	Integrated side column

production of an argon-enriched stream from a cryogenic air separation process		
EP 1 108 966: Process for separation of multicomponent fluids using a multizone distillation column	Air Products and Chemicals	Integrated side columns
EP 1 112 769: Verfahren zur Extraktivdestillation und Kolonne zur Durchführung des Verfahrens	Krupp Uhde	Integrated side column
DE 10 2005 003 499: Verfahren und Vorrichtung zur Trennung von Stoffgemischen	Linde	Integrated side column
WO 2004/103 937: Recovery of crude butadiene from a C4 fraction by extractive distillation in a partitioned column comprises controlling column parameters so that the bottoms stream consists of purified solvent	BASF	Dividing wall at upper end, butadiene
DE 10 2005 029 643: Verfahren zur Gewinnung von Reinstyrol aus einer Pyrolysebenzinfraktion	Uhde	Dividing wall at the upper end, styrene
CN 1745868: Extraction rectification tower has isolating wall vertically arranged from tower top to lower part of tower to divide internal space of tower into three sub-spaces	Jiangsu ind. college	Dividing wall at the upper end
DE 103 19 159: Verfahren zur destillativen Gewinnung von hochreinem Triethylendiamin (TEDA)	BASF	Dividing wall at lower end, TEDA
EP 1 329 449: Kolonne zur Aufkonzentrierung von Phthalsäureanhydrid	Lurgi	Dividing wall across the column, phthalic anhydride
US 4 230 533: Fractionation method and apparatus	Phillips Petroleum	Control
DE 35 22 234 : Verfahren zum energiegünstigen Betreiben einer Destillationskolonne	BASF	Control
DE 199 63 017: Verfahren zum Betreiben einer Trennwanddestillationskolonne	Wacker	Control
EP 0 780 147: Verfahren zur Regelung einer Trennwandkolonne oder einer thermisch gekoppelten Destillationskolonne	BASF	Control
US 6 551 465: Dividing wall column control system	UOP	Control
US 6 558 515: Dividing wall fractionation column control system and apparatus	UOP	Control
WO 2004/071 618: Control apparatus to control dividing wall section operation of dividing wall fractional distillation zone, has controller regulating input of heat in reboiler, and flow rate controller regulating the rate of net bottom stream	UOP	Control
DE 10 2004 004 530: Destillationsverfahren	Kao	Control
JP 2005 513 586: Distillation apparatus, e.g. for liquid slurry, has actuator to move movable perforated panel of	Kansai Kagaku Kikai Seisakusho	Control

change tray to adjust flow rate of steam supplied from one distillation column to other distillation column		
WO 2004/052 491: Improved distillation systems	Fluor	Control
DE 199 58 464: Verfahren zur Extraktivdestillation und Kolonne zur Durchführung des Verfahrens	Krupp Uhde	Control extractive distillation
Appr. 60 patents covering various fields of application	Various companies and institutions	Hydrocarbons, alcohols, phenols, ketones, aldehydes, esters, acetals, amines, amides, isocyanates, lactones, ethers, air separation and others

4.3 Institutes/companies working on the technology

(Provide the list of most important research centers and companies in Table 8)

Table 8. Institutes and companies working on the technology

Institute/Company	Country	Remarks
Technical University Delft (Dr. Olujic, Prof. Jansen)	The Netherlands	Construction issues
University Dortmund (Prof. Gorak, Prof. Kenig)	Germany	Reactive distillation
Montz, Hilden	Germany	Construction issues
BASF, Ludwigshafen	Germany	New fields of application

5. Stakeholders

5.1 Suppliers and developers

(Provide the list of key suppliers/developers in Table 9)

Table 9. Supplier and developers

Institute/Company	Country	Remarks
Montz	Germany	Manufacturer, delivered the majority of dividing wall columns, proprietary technologies
Koch-Glitsch	USA	Manufacturer
Sulzer	Switzerland	Manufacturer

5.2 End users

(Describe the existing and potential end-users, other than those already listed in Table 2)

Broader application in refineries, bio-ethanol plants, bio-diesel plants.

6. Expert's brief final judgment on the technology

(maximum 5 sentences)

The technology of dividing wall columns has been successfully introduced in chemical industry and is on the way to become a standard technology. Considerable economic advantages in the range of 30 % are provided with respect to energy consumption and investment costs. The future impact may be demonstrated regarding the 6 % contribution of chemical industry to world´s energy consumption, with 40 % of this amount relating to distillation processes.