Dr. Javier Tejero Salvador Departament Enginyeria Química i Química Analítica

Dra. Montserrat Iborra Urios Departament Enginyeria Química i Química Analítica



Treball Final de Grau

Levulinic acid upgrade to γ -valerolactone by hydrogenation by using a bifunctional catalyst

Eva Gutiérrez Campos

June 2020



Aquesta obra està subjecta a la llicència de: Reconeixement-NoComercial-SenseObraDerivada



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Quiero mostrar mi respeto, gratitud y agradecimiento a aquellos profesores que han formado parte de este trabajo, en especial a los Doctores Javier Tejero y Montserrat Iborra, sin vosotros este Trabajo Final de Grado no hubiera sido posible.

No quiero olvidarme de mis compañeros de laboratorio por el buen ambiente de trabajo durante todas las horas invertidas en él, y, en especial a Hazael Martínez por todos los consejos, guiarme y ayudarme durante la realización del trabajo.

Gracias a todos los profesores, des del primero al último por todos estos años.

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SUMMARY

Lignocellulose is one of the most abundant raw materials on earth. It can be found in plants, forest wastes among others, and several products widely used can be obtained from it. Some industrial processes are implemented with this aim. Biofine process allows the production of levulinic acid (LA) from lignocellulosic biomass. Levulinic acid is considered a platform chemical to produce several biochemicals and biofuels. γ-Valerolactone (GVL) can be obtained from levulinic acid in a single reaction.

In this project, some research about GVL synthesis from lignocellulosic biomass-derived levulinic acid has been done. The reaction of levulinic acid hydrogenation catalysed by a bifunctional ion-exchange resin loaded with palladium to produce γ -valerolactone has been carried out in a tubular fixed bed reactor. Kinetic and production studies were programmed but couldn't be performed because the exceptional coronavirus scenario cut short the project. However, the facilities were set up and some previous runs of the reaction were done to check if the reaction took place with the catalyst chosen.

Keywords: Biomass, Lignocellulose, Levulinic Acid, γ-valerolactone, Amberlyst CH28, Hydrogenation.

RESUMEN

La lignocelulosa es una de las materias primas más abundantes en el planeta. Se puede encontrar, entre otros, en plantas o restos forestales, y a partir de ella pueden obtenerse varios productos ampliamente usados. Con ese objetivo, algunos procesos industriales ya han sido implementados. Uno de ellos es el proceso Biofine, que permite producir ácido levulínico (LA) a partir de biomasa lignocelulósica. El ácido levulínco está considerado un "platform chemical" para producir productos bioquímicos y biocombustibles. La γ-Vlerolactona (GVL) puede ser obtenida a partir del ácido levulínco en una reacción directa.

En este proyecto se ha hecho búsqueda bibliográfica sobre la síntesis de la γ-valerolactona a partir del ácido levulínico derivado de biomasa lignocelulósica. Se ha realizado la reacción de hidrogenación de ácido levulínco para producir γ-valerolactona en un reactor tubular de lecho fijo utilizando una resina bifuncional de intercambio iónico cargada de paladio. Un estudio cinético y uno de producción estaban programados, pero no pudieron realizarse debido a que la excepcional situación provocada por el coronavirus acortó el proyecto. Sin embargo, la instalación se puso a punto y se pudo hacer algún experimento para comprobar que la reacción efectivamente tenía lugar con el catalizar elegido.

Palabras clave: Biomasa, Lignocelulosa, Ácido Levulínico, γ-valerolactona, Amberlyst CH28, Hidrogenación.

1. INTRODUCTION

Petroleum is a very important raw material used mainly in the production of fuel oils, commodities, and fine chemicals. Some decades ago, experts started to announce that the exhaustion of petroleum would be soon, because of the high international demand in front of the slow generation. It is not clear when the sources will be ended. There have been several theories about in which year the production of petroleum would be maximum, like Hubbert peak theory, in which M. King Hubbert estimated in 1956 that in the best-case scenario the peak oil production for the United States would be reached in 1970 (Black, 2014). By this year, technologies were improved, and new sources were explored, so the production greatly surpassed the amount estimated. It is also true that the production decrease since that year, but very slowly, so besides the fact that the production was higher than predicted, the slow decrease of the curve involves that the exhaustion was not as soon as expected. Since that year, the global peak has been estimated and surpassed again. It might make us thought about if the petroleum really will be exhausted someday. But besides the exhaustion, there is an issue that nowadays compels the most to replace petroleum: the combustion of petroleum products is harmful to the environment.

In general terms, the consumption of fossils fuels has been stabilised, but in the transportation sector, which is the most petroleum demandant, the requirement is increasing, and with the development of the society, it is supposed to continue rising, as seen in **Figure 1**. Therefore, it is necessary to find alternatives to reduce the use of petroleum sources.



Figure 1 Oil consumption by sector in the world(International Energy Agency, n.d.)

The main challenge nowadays is replacing fossils fuels economy by one based on renewable sources that provide economic, social, and environmental benefits. In 2002, a substitution of 25% of fossil fuels with renewable sources in 30 years was intended (Willke and Vorlop, 2004). However, in 2017, biofuels only represented 3% of the energy consumed in the transportation sector (International Energy Agency, n.d.). Some suggestions have already been tried, and some of them have already been implemented. Two of the most developed alternatives to replace petroleum in the transport sector are electric cars and biomass.

1.1. ELECTRIC VEHICLE

The electric car is one of the most promising alternatives to avoid fossil fuels. In the 19th century, even before the gasoline car was invented, a prototype was already developed to replace steam vehicles. Between the 1880s and 1920s, the electric car experimented a development period very intensive and build up the basis of the electric car we know nowadays. The battery they used at the beginning was a lead-acid battery. The batteries were improving while electric cars, so in 1901, the nickel-iron battery was developed, and allowed to store 40% more energy than the lead-acid battery (Høyer, 2008). Electric cars started to be commercialised at the beginning of the XX century. At that moment, the autonomy that those cars had was enough to move around the city, but the high cost of the used batteries made the electric cars only available for a few rich people. Some years later, used cars fell in disuse because of the connections

between cities improved, and the electric car couldn't travel big distances. Several upgrades were introduced during the XX century in electric cars, such fast battery swapping systems or using the electric drive motor himself as a generator to recharge the battery in certain moments of driving. known also as regenerative braking technologies. In a few years, the hybrid car combined both gasoline and electric motor, and permit travelling out of the cities with no worries about the battery, but it was unaffordable and didn't go further. Electric cars have been in a continuous up and down, especially depending on the availability of gasoline, related to social aspects, like wars. In general terms, only in the period of a gasoline shortage, the electric car has been more used. During the second half of the XX century, environmental awareness becomes more important, and the discussion about renewable and non-renewable sources to produce energy became a general interest, mainly in the transportation sector. The issue of petroleum exhaustion and the high levels of air pollution on account of vehicle emissions among others, bring into focus the electric vehicle again. Many governments promote initiatives to stimulate the use of the electric car and introduced restrictions to reduce those levels of pollution. But even though electric cars have been improved, the same problems that those types of vehicles had at the beginning, are still detrimental to reach the objective of developing an electric car able to compete with gasoline and diesel cars. The autonomy achieved in the electric cars nowadays is about 350 km at the most but it is not enough to travel long distances, and the convenience of refuelling an oil car in front of having to recharge a battery beside the price, still makes difficult the change. Hybrid cars can overcome the limitations of autonomy because they combine both energy sources, electricity, and fuel. During urban driving, hybrid cars use the electric motor (with several stops that allows the battery to be auto-recharged if the car uses regenerative braking technologies) with no pollutant emission, but during extra-urban driving, when the electric motor has been discharged the fuel combustion is necessary, and due to the hybrid car is heavier than a similar conventional model, the consumption is higher (INSIA-UPM, 2019). So, if they are used in urban areas, they are a good alternative, but if they are mostly used in interurban areas, the problem not only is not solved with those cars but can worsen. Moreover, the electricity used both in electric and hybrid cars often come from nuclear or fossil sources (Høyer, 2008), so it may be nonsense. It is also

important to be aware that increasing the affordable distance without having to recharge involves that batteries must be bigger. In most of the electric vehicles produced nowadays, the battery used is made from rare earth elements (Lopez Gómez and Rodríguez Largo, 2019). Those

elements, apart from being difficult to obtain, are difficult to be recovered, so it is a pending matter knowing how to do it, considering that it has only been tried on a small scale. So, when all batteries used in the vehicles need to be removed, if there is no way to recycle them, it will be unsustainable. Hence, although electricity is one of the most hopeful alternatives to solve the fossil fuel issue, it is not the complete solution, at least as we know them today. **Table 1** summarises the most important advantages and disadvantages of the electric vehicles nowadays.

Table 1 Advantages and disadvantages of electric vehicles

	Advantages		Disadvantages
-	Can avoid fossil fuels	-	Not enough autonomy
-	0% pollutant	-	Electricity obtained from non-renewable
	emissions		sources
		-	Contaminating batteries
		-	Long charging time in front of the quick
			refuel of the gasoline car

1.2. BIOMASS

Biomass consists of materials that are or have been part of a living organism. In terms of energy, organic material related to plants is included. There are many ways to convert biomass into energy, so it could replace petroleum in different processes. Biomass is suitable to produce biofuel and the fact that its raw material is biological, and it is quick to regrow, makes it a good alternative for fuel oil. One of the principal advantages of using biofuels from the biomass is that the CO₂ produced with the biofuel combustion had been previously absorbed by plants in the growing process, avoiding neat CO₂ emissions that cause the greenhouse effect (Fernández, 2003). The energy produced from the biomass can be considered renewable because its energy contents come from solar energy fixed by photosynthesis. As seen in **Table 2**, the carbohydrate structure of biomass makes it more oxygenated compared with fossil fuels. Since 2005 in Spain, commercial gasoline must have less than 5% of sulphur (Fernández-Feal et al., 2009) to reduce the environmental effects of pollutant emission, so it is important to consider that biomass has less than 1% of nitrogen, sulphur, and chlorine.

Component	wt% of dry matter	
Oxygen	30-40	
Carbon	30-60	
Hydrogen	5-6	
Nitrogen, Sulphur, Chlorine	<1	

Table 2 Biomass composition (Jenkins M. et al., 1998)

Depending on the origin of the biomass and the technology used to produce the biofuel, they can be called (Aro, 2016):

- First generation biofuels
- Second generation biofuels
- Third generation biofuels

First generation biofuels are obtained from edible biomass, like sugar, oils, and cellulose. Crop plants are grown to produce biofuel, but since they use food crop plants that can be destined to the food supply chain, it poses an ethical problem. Hence, although the first generation fuels can replace fossil fuels, another alternative should be considered.

Second generation fuels are those which come from feedstocks of lignocellulosic non-food material. Lignocellulosic is a component found in straw, crop plants grown in marginal lands, and even in forest waste. Food crop plants can also be used as well, but only once they are no longer useful for the food sector. By using this type of biomass, the ethical problem caused by using edible biomass is solved. In 2007, in Spain, 25.584.000 tons of municipal solid waste was collected, and as shown in **Figure 2**, 44% was organic waste. Nowadays, the principal waste management is letting them in landfills, so if part of the organic fraction could be used to produce second generation biofuels, it would suppose that these residues that are underutilized could be worthy again. But second generation biofuels only would be able to displace, at most, a third of the petroleum consumed by the transportation sector (Serrano-Ruiz and Dumesic, 2010).



Figure 2 Municipal Solid Waste collected in Spain in 2007 (Grau and Farré, 2011)

Moreover, because of the treatments needed to convert lignocellulosic biomass into liquid biofuels are more difficult than the ones requested in the process of first generation biofuels, the production costs are higher.

Third generation biofuels are produced by algal biomass. They are still being invested, and by this moment, algae are not being used to produce biofuel on a large scale.

Even biomass is a promising alternative, some issues can hinder its extended use. One aspect that must be considered is that not all biomass has the same properties, because depending on where it comes from, its composition is different. In **Table 2**, the typical composition of biomass is shown. It could pose a problem at the design of the production process. It is also important, that its availability is not constant, and logistics may suppose an increase in the processes cost. Furthermore, if all the petroleum requirements were covered by biomass, it would be unsustainable and soon the biomass would be exhausted.

As mentioned before, the amount of biomass is not boundless. **Figure 3** compares the energy consumption and supply in the world since 1990 to 2017. Oil and biofuel are represented, and the supply of both is always over the consumption. If consumption grows, the supply grows too, so the theories about the exhaustion of petroleum are again questioned. When more production is required and the infrastructure or sources are not enough, the industry invests to find new ways to supply it. According to the graphic, it is also evident, that the supply of biofuels is much lower

than oil consumption, so, with the accessible technologies, biomass available is far from being able to replace the demand of fossil fuels of the world.



Figure 3 Energy supply and consumption of the world (International Energy Agency, n.d.)

Since biomass available can't supply biofuel enough to forget fossil fuels, a reliable alternative is combining both. Some biomass derivates can be used as additives to gasoline. Levulinic acid (LA) is one of them. It is a product derived from cellulose and can be obtained easily from lignocellulosic biomass. LA and its derivates can be useful in very different fields, so it is considered a worthwhile platform chemical to be investigated. Due to its reasonable price and its potential uses in the biorefinery industry, LA is one of the top 12 most promising building blocks according to the United States Department of Energy (Pileidis and Titirici, 2016). By hydrogenation of LA, it is possible to obtain γ -valerolactone (GVL), an intermediate that has been considered a good blending of ethanol in gasoline (10%v/v) (Alonso et al., 2010). The percentage of blending is important considering that European standards allow only 5%v/v ethanol blends (Serrano-Ruiz and Dumesic, 2010), so it could be interesting to continue investigating GVL.

2. OBJECTIVES

This project aimed to upgrade LA to GVL by hydrogenation catalysed by a bifunctional ionexchange resin loaded with palladium. The objectives were:

- Update bibliography related to reactions, primary and secondary ones, useful catalysts, and conditions.
- Setting up an existing experimental installation.
- Checking the viability of the catalyst, its activity, and its selectivity.
- Establishing the experimental operation conditions
- Perform a preliminary kinetic study

Due to the situation caused by the COVID-19 pandemic and facing the impossibility of doing all experimental work, the project has been adapted.

3. LEVULINIC ACID

4-Oxopentanoic acid, also known as levulinic acid, is a crystalline keto acid derived from cellulose. Its reactivity is relatively high, so it is promising to be an intermediate to other valuable products. It is soluble in water and polar organic solvents. Its melting point is 33°C, so it is usually solid at room temperature but can turn into a liquid with not much heating.



Figure 4 Levulinic Acid

Before processes to obtain LA from biomass were developed, it was obtained by a chemical process with many bottlenecks such as the high cost of raw materials, the low yields reached because of undesirable side reactions, and difficulties related to recovery product and catalyst. Therefore, even it was valuable, not only as a product but also as a precursor to other products as it will be seen later, it was not enough profitable. Since its production from biomass is available, LA has reduced the production cost making its commercialization easier and becoming a reliable platform chemical for fossil fuel substitution.

The different intermediates and products that can be obtained from lignocellulosic raw material are shown in **Figure 5**.



Figure 5 Products from lignocellulose (Rackemann and Doherty, 2011)

3.1. LEVULINIC ACID FROM LIGNOCELLULOSIC BIOMASS

Lignocellulose is a structural element of plants, both edible and inedible. Non-food lignocellulose can be obtained from agricultural residues, forest waste, agriculture residues, or municipal paper waste. The three main components of lignocellulose are cellulose (38-50%), lignin (15-30%), and hemicellulose (23-32%) (Pileidis and Titirici, 2016). It is abundant and cheap and has widespread worldwide availability.

Technology	Energy Requirement	Cost	Technology Status
Biofine Process	High	Medium	Semi-commercial
Homogeneous catalysis	Medium	High	Semi-commercial
Heterogeneous solid acid catalysis	Medium	Medium	Research
Heterogeneous metal catalysis	Low	Medium	Research
Solvolysis	High	High	Research
Ionic liquid	High	High	Research
Supercritical fluids	High	High	Research

Table 3 LA production technologies (Morone et al., 2015)

There are different technologies to obtain LA from lignocellulosic biomass as seen in **Table 3**, but only the Biofine process is used at a semi-commercial scale.

LA can be obtained from lignocellulose by Biofine process using homogeneous catalysis, in ranges of 0.42-0.595kg LA/kg cellulose (Hayes et al., 2006). Conversion of lignocellulosic biomass into LA needs a pretreatment. The lignin that covers cellulose and hemicellulose must be removed to allow them to react. This pretreatment usually consists of the breakdown of lignocellulosic biomass complex, removing lignin in a way that can be reused, decrystallising the cellulose fibers, and reducing the mass transport limitation for catalysts. Depending on which type of biomass is been treated, this pretreatment will be easier or more difficult, increasing the cost. Pretreatment supposes around 20% of the total production cost (Morone et al., 2015), so it is important to choose the most efficient treatment. Biofine process consists of a tubular plug-flow reactor fed with the lignocellulosic biomass (hexose sugars) mixed with a mineral acid agueous solution like diluted sulphuric acid (Galletti et al., 2012). Steam is also injected into this first reactor to hydrolyse the biomass feedstock for a short time (few seconds). The outflow of this reactor with the product formed in this reaction (HMF, 5-hydroxy methyl furfural) is fed to a continuously stirred tank reactor, where it is hydrolysed for 15 to 30 minutes. The second reactor gives LA as a product (around 60% conversion of cellulose (Bozell et al., 2000)), and formic acid as a by-product. If there is hemicellulose in the raw material, furfural is also obtained as a by-product. Formic acid and furfural are recovered from the second reactor from the vapor stream, whereas LA is removed as a liquid and filtered to separate it from the humins formed. The process is operated in continuous and at temperatures close to 200°C (higher in the first reactor and lower in the second). Formic acid can be discomposed into H_2 , CO, and CO₂ at temperatures near to 200°C, so it might not be in the proportion expected. Figure 6 shows the Biofine process flowchart.



Figure 6 Biofine process (Girisuta and Heeres, 2017)

Although the Biofine process is the nearest process to be industrialized due to its high yields, the recovery of LA from the solution resultant is not easy and the humins obtained (not big amount due to the reactor and the conditions are already designed to avoid them as much as possible) pose a clog problem to the system. Moreover, the energy and water requirements are high.

Homogeneous acid catalysts catalyse the hydrolysis reaction. The LA yield can vary depending on the acid used, the reactor design, operating conditions, and the pretreatment done to lignocellulosic biomass. In order to get the highest LA yields, these factors must be optimised, and the optimal conditions will be different according to biomass composition. Therefore, scaling this method from laboratory to industrial plant has been limited. Apart from scalation limitations, it has other problems such as the recovery of the catalyst and environmental and corrosion issues caused by the acid.

The advantages of using heterogeneous acid catalysts (acid and metal) in front of homogenous ones are the easier recovery of the catalyst and avoiding corrosion problems despite the higher selectivity. But the use of this technology needs to much research since LA has strong adsorption on the catalyst and this decreases the LA yields (Morone et al., 2015).

Another technology available to LA from lignocellulosic biomass is solvolysis. It has been tested with water or organic solvents, but it already needs to be more investigated to find the ideal solvent which increases the yield with no environmental impact. This technology consists on using the solvent as a reactant. It must be in higher proportion in front of the other reactant, so it involves

large volumes of the solvent and complicates the recovery of LA. In contrast, the pretreatment of biomass needed in other technologies is not necessary.

lonic liquids are liquid salts than can be used as a solvent and as a catalyst. In the LA production, they act as a catalyst. They are worthy because of their thermostability, the easier separation from the product, their low vapor pressure, and the mild reaction temperatures. This technology has been studied mostly in the production of HMF, but the same can be applied to LA production. However, those liquids are expensive and need to be purified, which is not easy and limits their industrial use.

Supercritical fluids are another type of solvents with acidic and basic properties that favour the product recovery avoiding by-products formation. On the other hand, this technology needs high pressure, so the equipment and the operational costs are high.

3.2. LEVULINIC ACID DERIVATES

It is considered a platform chemical useful in different areas. Its derivates can replace lots of chemicals that come from oil. In **Figure 7**, there are some of them. Although many of these products are not convenient to be commercialised on a large scale, because of the production cost, LA has become a reliable alternative to produce biofuel and fuel additives from a renewable source.



Figure 7 LA products (Rackemann and Doherty, 2011)

The three most developed products converted from LA are methyltetrahydrofuran (MeTHF), δ -aminolevulinic acid (DALA), and diphenolic acid (DPA). These three products, specially MeTHF and its precursor GVL would permit the expansion of the LA market. Furthermore, economic experts predicted that depending on the scale of the operations the production costs could be down to 0,04-0,1\$/lb (Bozell et al., 2000), so it would help to its expansion.

Table 4 Potential LA market (Bozell et al., 2	.000)
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Product	Use	Potential LA Market (million lb/year)	
MeTHF	Fuel extender, fuel additive	10 000 – 100 000	
DALA	Biodegradable herbicide / pesticide	175 - 350	
DPA	Monomer, substitute for bisphenol A in polymer manufacture	35	

3.2.1. MeTHF

MeTHF most extended use is as a transportation fuel extender because it is miscible with gasoline and hydrophobic. It can be used to reduce other components in gasoline to meet the fuel

requirements stipulated to recover environmental issues, without any adverse functioning. The percentage of blending in a gasoline mixture is not great (around 1%v/v) (Bozell et al., 2000) but considering the amount of gasoline used in the world, this little percentage of blending might result in a big demand of MeTHF at the end of the year. In **Table 4**, the potential market of each of the three most interesting products derivate from LA is shown, and MeTHF is considerably high. Obtaining MeTHF directly from LA is difficult, but it can be easily derivate from GVL. More about MeTHF will be explained later, in the chapter dedicated to GVL and its uses.

3.2.2. DALA

DALA is a product widely used as an herbicide with high activity towards dicotyledonous weeds and little activity toward monocotyledonous. The activity is comparable to other commercial herbicides with the advantage that it is completely biodegradable. It is also used as an insecticide. It has also been found that DALA is suitable as a component in photodynamic therapy as a treatment of cancer.

There are several routes to the synthesis of DALA from LA at a laboratory scale, but they are not convenient to be introduced at a large scale. Some approaches have been tested but with many issues. The most common is a multistep process in which the first steps provide relatively low yields, and by producing DALA, a large amount of wastes is also produced. Even though, in general none of these approaches are feasible due to the multiple steps needed, the high cost of the raw material and the toxicity of the intermediates involved in the processes.

3.2.3. DPA

DPA is commonly used in the production of materials such as polymers. Its applications are similar to other products but DPA was more expensive, so it was less commercialised. Since LA is reducing its production cost, DPA is becoming a suitable material to produce polymers from a renewable source and to replace products like bisphenol A, which is forbidden because it is dangerous for human health.

There is some research about other possible uses of DPA like fire retardants or non-toxic marine coating. Other possible applications of DPA are paints, medicines, and adhesives among others (Bozell et al., 2000).

4. **Y-VALEROLACTONE**

GVL is a cyclic ester with 5 atoms in the ring. In ambient conditions, it is a colourless stable liquid with a high boiling point. It discomposes neither with time nor in the presence of water or oxygen. It has been considered non-toxic and its main risk is flammability, but its volatility is low and makes the flammability risk of GVL at ambient conditions low. It doesn't form peroxides in air.



Figure 8 GVL

When GVLS is produced from cellulose, the intermediates obtained (glucose, HMF, LA, and formic acid) are miscible with water. This makes the biodegradability of GVL easy. Another benefit of obtaining GVL from cellulose is that almost all dangerous, halogenated, or phosphorous compounds produced in its synthesis from chemicals are not produced. Chlorine is the only compound that can remain. Chlorine is involved in the reaction when the production of LA is catalysed by HCI. Formic acid can be corrosive, but usually, it is internally discomposed into CO₂ and H₂. CO₂ is easily converted into other valuable chemicals and H₂ can be used in the hydrogenation of LA to obtain GVL. The commercialization of GVL is not widely extended due to its production cost. Nevertheless, with the improvement of LA production, it is expected that GVL production would be more affordable.

4.1. GVL USES

As said before, GVL is one of the most promising products obtained from lignocellulosic biomass since its possibilities to displace petroleum derivates such as gasoline, fuel additives, or polymer precursors. It has excellent properties as a solvent, and it is a good precursor for high-value chemicals and fuels. Besides the uses related to fuels, its sweet and herbaceous smell makes it appreciate in the production of perfumes, and it is widely used as a food additive. In

Figure 9, there are represented different products that can be derivate from GVL and its obtention pathways. Some of its most promising uses are explained below.



Figure 9 GVL derivates (Alonso et al., 2013)

4.1.1. GVL as a solvent

GVL can be used as a solvent, and precursor to other green solvents, in processes of obtention of different valuable chemicals from lignocellulosic biomass. **Figure 10** shows some of these products obtained using GVL as a solvent, and the most relevant are explained below. After studying GVL, Phillip G. Jessop concluded that it has advantages in front of other common solvents (Jessop, 2011).



Figure 10 Lignocellulosic derivates obtained using GVL as a solvent (Alonso et al., 2013)

GVL can be used as a solvent in the production of HMF, GVL, and LA from fructose with advantages. In the production of GVL, GVL is used to dilute LA with the main benefit that the product and the solvent don't need to be separated since they are both GVL. When the solvent is used in the production of LA or HMF, some aspects must be considered, especially product purification. Even though, some research has been done and has found that there are processes where the product and the solvent don't need to be separated, for example, the production of 2,5 dimethylfuran from HMF can be done without separating GVL, which can be separated from the final product easier. Even some solutions have been proposed, the catalyst separation is still an issue that needs to be fixed.

It also has been used in the production of HMF from C₆ sugars. The main advantage is that high yields can be obtained using a monophasic system and solid acid catalyst thus mixing, separating phases and, catalyst recovery issues are solved.

GVL can also be used as a solvent to convert hemicellulose into furfural. It solubilised the degradation products formed, and the reaction is faster. It also reduces furfural degradation,

increasing furfural yield in monophasic systems. In this case, the separation between GVL and the products can be easily made by distillation.

Using GVL to deconstruct biomass makes it easier to produce LA from cellulose. The reaction time and the catalyst needed decrease, and the furfural generated becomes more stable. The furfural is converted into furfuryl alcohol by hydrogenation while LA is converted into GVL. The furfuryl alcohol is later converted into LA catalysed by an acid catalyst and hydrogenated again into GVL. This process improves the use of lignocellulosic biomass in GVL production and doesn't need a separation step.

4.1.2. GVL to produce polymers

Biopolymers are those polymers made from biomass totally or partially. The research of biopolymers is increasing because of the different material properties of these polymers compared with the ones derived from petroleum. Moreover, the process to produce polymers from biomass is more suitable.

Starting from GVL, different ways to produce polymers have been studied. One of them is the ring-opening of GVL to methyl pentenoates with yields of around 98% (Yan et al., 2015). The difference between its volatility and that of GVL makes the separation easy. Methyl pentenoates can be converted into different nylon precursors. By adding formaldehyde to GVL, it is possible to produce α -methylene- γ -valerolactone with selectivities over 95% (Manzer, 2004), which is a monomer with similar properties to methyl methacrylate but with improved thermal stability. These two routes are represented in **Figure 9**.

It is also being studied GVL ring-opening with amine compounds. The products obtained (y-hydroxy-amides when the reaction takes place at mild conditions) can be used to produce a green polymer. **Figure 11** shows different polymer precursors obtained by this method. By condensation of GVL and different monomers, new polyurethanes can be obtained. Because of the differences in the chemical structure of polyurethanes precursors, the range of thermal properties of the final products is wide. So, by varying the structure of the amines, it is possible to produce many promising polymers.



Figure 11 Polymer precursors from GVL (Yan et al., 2015)

Anyway, for the moment, the synthesis of polymers derived from biomass is just being studied at laboratories with low yields, thus more research is needed to decide which processes are suitable to produce on a large scale.

4.1.3. GVL uses related to fuels

GVL can be used as a liquid fuel but due to its high water solubility, it has not been tested as a pure fuel. Whereas it is a profitable additive to petroleum fuels. It has been proved that for mixtures with the same percentage of GVL or ethanol (10%v/v), the one with GVL has lower vapor pressure than the one with ethanol, improving the combustion at similar octane numbers. **Table 5** shows some properties of this 95 octane gasoline the two blended gasolines. Although GVL is interesting mostly as a fuel additive, it can also be converted into other products interesting as fuels. Some GVL derivates with theses uses are explained below.

	AN-95 gasoline	90%v/v AN-95 + 10%v/v EtOH	90%v/v AN-95 + 10%v/v GVL
Density (15ºC) [kg/m ³]	733,5	737,8	765,8
Oxidation stability [min]	ОК	ОК	ОК
Peroxide number [mg/kg]	1,4	1,72	
Vapor pressure (Dry vapor pressure equivalent) [kPa]	54,6	65,1	56,6
Vapor pressure (Air saturated vapor pressure) [kPa]	63	71	62,2
Evaporated up to 70ºC [%v/v]	27,2	47,9	24,1
Evaporated up to 100ºC [%v/v]	52,3	57,3	46,2
Evaporated up to 150°C [%v/v]	90	90,7	80
Final boiling point [ºC max]	181,9	181,6	202,2
Distillation residue [%v/v]	1	1	0,9
Gum content blown/mg per 100mL	1,6	1,9	
Existent gum washed/mg per 100mL	0,5	0,8	0,8
Copper strip	1A	1A	2a
Motor octane number	88,8	89,3	89,2
Research octane number (blending RON)	97,2	97,4 (105)	97,3 (105)

Table 5 Properties of 90% 95 octane gasoline and 10% EtOH or GVL (Horváth et al., 2008)

MeTHF

By using MeTHF as a fuel additive, some limitations related to the transport sector such as blending limits, low energy density, or corrosive problems that other products cause can be partially overcome. As an example, the 10%v/v of blending with ethanol achieved with GVL can be upgraded up to 70%v/v (Alonso et al., 2010). The mileage from fuels blended with MeTHF is similar to petroleum fuels due to its properties, so there are no disadvantages in that aspect. Moreover, blending MeTHF with ethanol in gasoline helps to reduce the vapor pressure of ethanol.

MeTHF is obtained by hydrogenation of GVL and the subsequent dehydration (Figure 12), and high yield up to 90% can be reached (Yan et al., 2015). The main challenges related to its
production is the yield of coke when a heterogeneous catalyst is used and find the reaction parameters that optimize the process.

MeTHF can also be converted into fuels and its solubility makes it suitable to replace solvents to obtain higher reaction temperatures or easier product separation. Even MeTHF and GVL cannot be used as jet or diesel fuels, their limitations can be solved with some processes that are being studied and will be explained later (hydrocarbon fuels).



Figure 12 MeTHF from GVL (Yan et al., 2015)

Hydrocarbon fuels

There are several ways to produce liquid hydrocarbon fuels from GVL, but two of them are the most common and will be explained referring to **Figure 13**, where the different steps are numerated from 1 to 10.

The first one consists on produce pentanoic acid (step 1) and then butenes (step 2). By oligomerization and hydrogenation of these butenes C_8 and C_{12} -alkenes are obtained (steps 3 and 4).

The other route upgrades GVL producing pentanoic acid through ring-opening on acid sites and hydrogenation on metal sites (step 5). Acid pentanoic is ketonised to 5-nonanone (step 6) which is then hydrogenated to produce 5-nonanol (step 7). From 5-nonanol 3 different products can be obtained: C₉-alkanes are obtained by dehydration and hydrogenation (step 8), branched C₉-alkanes are obtained by dehydration, isomerization, and hydrogenation (step 9), and C₁₈alkanes are obtained by dehydration, oligomerization, and hydrogenation (step 10). It is important to know that the most important steps to achieve high yields of final products are the obtention of pentanoic acid and 5-nonanone (steps 5 and 6). Nonane can be produced by hydrogenation of a mixture of linear C₉-alkanes and can be used as an additive to diesel fuel or converted into gasoline. In **Figure 9**, the uses of these products are shown.

Even the advantages, the production of alkanes still requires being studied in order to find how to increase the yields and reduce the needed steps.



Figure 13 Hydrocarbon fuel from GVL (Serrano-Ruiz and Dumesic, 2010)

Valeric esters

Valeric esters are promising to use them as oxygenating fuel components for gasoline and diesel fuels. Long carbon chain esters are suitable for diesel while short carbon chain esters are used in gasoline blending. The ignition properties and flow characteristics of valeric esters are compatible with diesel fuel and producing biofuels from valeric esters improves biofuel performance compared with biofuels produced from other alternatives proposed such as ethanol, GVL or MeTHF (Zhou et al., 2018).



Figure 14 Valeric esters from GVL (Yan et al., 2015)

Starting from GVL, the synthesis of valeric esters consists on ring-opening GVL to pentenoic acid and then hydrogenating it to pentanoic acid which is esterified with alcohols or glycols to produce the corresponding valeric ester in presence of a bifunctional acid-metal catalyst (**Figure**

14). It has been proved that with the right catalyst and conditions, it is possible to obtain yields of 90% of pentanoic acid continuously for more than 1500h (Alonso et al., 2013).

Several processes to produce valeric esters are at the begging of research and they aim to reduce the metal content of the catalyst without worsening the selectivity.

5. HYDROGENATION OF LA TO PRODUCE GVL

GVL can be produced by hydrogenation and dehydration of LA. If LA is hydrogenated first, 4hydroxypentanoic acid, also known as γ-hydroxyvaleric acid, is formed as an unstable intermediate which is then ring-closed by intramolecular esterification and loses water molecule spontaneously, forming GVL. The hydrogenation is catalysed by a metal-catalyst while the esterification is acid-catalysed. If LA dehydration takes place first, the intermediate formed is αangelica lactone (AL) which is hydrogenated into GVL. This second pathway also needs an acid and a metal functionality and GVL yields achieved are lower than with the first one because of the formation of coke from AL catalysed by the acid present. AL is formed at temperatures around 573-623K (Serrano-Ruiz et al., 2010) and can be avoided by working at mild temperatures or by using a non-acid catalyst. It is interesting to perform the reaction under the first pathway due to the coke formed in the second one can be deposited on the acid surfaces of the catalyst avoiding its good performance. In our process, the operating temperature must be below 403K as will be seen (Chapter 6), so AL formation won't take place. **Figure 15** illustrates both processes and the upgrading of GVL to 5-nonanol.



Figure 15 Reaction pathway from LA to 5-nonanol (Serrano-Ruiz et al., 2010),(Alonso et al., 2010)

5.1. **EXPERIMENTS REPORTED**

(Fu et al., 2016) hydrogenated LA to produce GVL using as a catalyst Ni/Al₂O₃. They first perform some experiments to determine the more suitable Ni load and solvent for this reaction. They tried with water and dioxane as a solvent and with loads between 10% and 40%. They conclude that the best results were obtained with 40% of Ni load using dioxane as a solvent. They operate in a 100mL batch reactor with a stirring speed of 1000rpm, at 180°C. They filled the reactor with 1g of LA. 0.1g of Ni/Al₂O₃ (40%), and 40mL of dioxane at 3MPa hydrogen. They let the reaction proceed during 2h. Then they filtered and washed the reaction mixture and diluted with deionised water to 250mL. They analysed the composition using an HPLC (High Performance Liquid Chromatography). The LA conversion reached was 100% with 99.2% of selectivity to GVL. They also studied the influence of the temperature, the pressure, and the S/C (substrate/catalyst) ratio. Rising temperature from 150°C to 180°C increased the LA conversion without modifying the GVL selectivity. They found that LA conversion grew when the pressure changed from 1MPa to 2MPa, but at 3MPa or high, it remained approximately constant. S/C ratio had an important influence, by raising from 10 to 100, the LA conversion dropped from 80% to 20% (experiment conditions 180°C, H₂ pressure 3MPa, LA load 1g, and reaction time 1h). GVL selectivity also decreased, but not as much as LA conversion. Ni has been used by many researchers due to its high activity in the upgrading of LA into GVL and its high selectivity towards GVL (Dutta et al., 2019)

(Piskun et al., 2016) studied the reaction on different Ru based catalysts. They used deionised water as a solvent an operate in a packed bed reactor with a catalyst load of 2g. They carried out the experiments at the bench conditions shown in **Table 6**. They reduced the catalyst (Ru/C 5wt%) for 4h at 350°C, 20bar, and 250mL/min of reducing gas (10%v/v H₂ + 90%v/v N₂) and determined that for the same conditions the conversion dropped from 95% to 75% if the catalyst was previously reduced, so they didn't activate it before their experiments.

LA inlet concentration [mol/L]	1,1-1,2
LA feed rate [mL/min]	1
H ₂ feed rate [mL/min]	30
Temperature [°C]	90
H ₂ pressure [bar]	45
WHSV [g _{feed} /g _{cat} ·h]	30
Time on stream [h]	6

Table 6 Bench Conditions

They measured the liquid temperature at the reactor exit, and when it was 85°C, they considered the reaction started. Every 15min samples of the reaction mixture were analysed by H MNR (proton nuclear magnetic resonance) to determine the composition. It was observed that after 1-2h the reaction reached the steady-state, and the conversion achieved for every catalyst is summarised in **Table 7**. They also studied the influence of the LA feed concentration (with Ru/C 5wt% and bench conditions **Table 6**) and observed that for LA conversion was only 5%. They carried out other experiments to improve LA conversion at 11,2M LA concentration: they modified some of the bench conditions increasing the temperature to 150°C, increasing the H₂ feed rate to 120mL/min and using 5g of catalyst instead of 2g, and LA conversion achieved was 52% in front of the 5% reached with bench conditions.

Catalyst	Ru loading [wt%]	LA conversion [%]	GVL selectivity [%]
Ru/C	0,5	92	78
Ru/C	2	96	63
Ru/C	5	98	83
Ru/ɣ-Al ₂ O ₃	0,3	31	31
Ru/ɣ-Al ₂ O ₄	0,5	26	62
Ru/TiO₂	1	26	54

Table / R	u catalysts
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(Serrano-Ruiz et al., 2010) also studied the hydrogenation of LA into GVL on Ru/C (5wt%) at 150°C and 35bar, with a WSHV about 4,8h⁻¹ and an aqueous solution 50wt% of LA. They

obtained 90% of LA conversion and 96% of GVL selectivity. The deactivation of the catalyst was also studied, and they observed that after 106h of reaction LA conversion only decreased from 90% to 68%, and after re-hydrogenated for 2h at 400°C, the activity of the catalyst was partially recovered, reaching initial conversion of 83% when the catalyst was reused.

(Piskun et al., 2018) tested the reaction with different supported Ru catalysts using water or dioxane as a solvent and found that Ru/TiO₂ had the best performance. Parr batch autoclave reactors were used for their experiments. They considered the reaction had started once the reactor reached the temperature desired. The experiments where LA was diluted with water were in a 100mL reactor, using 2,9g of LA in 40mL of deionised water and the composition of the resultant mixture was analysed by H NMR. In those where the solvent was dioxane the reactor volume was 50mL, 2,2g of LA were diluted in 28mL of solvent and the analysis of the composition of the experiments are shown in **Table 8**. After 6h, in the reaction with water, full conversion of LA was obtained with 89% selectivity to GVL and 4-hydroxypentanoic was also formed as a by-product. Whereas the reaction with dioxane almost reached full conversion after 4h but with 100% GVL selectivity.

	<u>S</u> olvent	
	Water	Dioxane
LA feed concentration [LA]	0,63	0,64
Solvent intake [mL]	40	28
Temperature [°C]	90	150
Hydrogen pressure [bar]	45	45
Catalyst intake [g]	0,06	0,06
LA/Ru molar ratio	4350	3200
Stirring rate [rpm]	2000	1250
Reaction time [h]	4	4

Table 8 Reaction conditions with Ru/TiO2 catalyst in batch

(Moreno-Marrodan and Barbaro, 2014) used bifunctional metal/acid heterogeneous catalyst to perform the reaction in a continuous reactor. They immobilized Ru nanoparticle onto a sulfonic ion exchange resin (DOWEX 50WX2-100 gel-type resin). They used a gas chromatograph to analyse the reaction mixture compositions.

They first operated in a batch reactor (25mL) stirred at 150rpm with LA concentration 0,43M in water, 60mg of catalyst (loaded with 0,68wt% Ru), and S/C molar ratio of 420. They carried out different experiments to analyse the influence of temperature and H₂ pressure and found that in those conditions, at 70°C and 5bar after 7h, full conversion was achieved with more than 99% GVL selectivity. They also experimented with non-acid support to observe the positive contribution of acidic resin: at the same conditions mentioned before, with non-acid support the LA conversion after 4h was 16,2% in front of the 79,5% using a bifunctional catalyst.

Then they used a 176µL reactor and packed 40,1mg of the same catalyst used at the batch reactor experiment. The LA feed was 0,02M in concurrent flow with H₂ at different flow rates operating at 70°C. It was shown that with 0,15mL/min of LA solution and 1mLl/min and 5bar of H₂, GVL selectivity was almost 100% and LA conversion higher than 90%. They found that conversion could be raised to 100% keeping the selectivity by decreasing the solution flow rate to 0,10mL/min (0,7mL/min H₂ at 4,8bar) or by increasing the hydrogen flow rate to 1,5mL/min at 5,3bar (0,15mL/min LA solution).

To conclude two tables summarise the experiments reported above. **Table 9** for experiments performed in a batch reactor and **Table 10** for the ones performed under continuous flow.

	(Fu et al., 2016)	(Piskun et al., 2018)		(Moreno-Marrodan and Barbaro, 2014)
Catalyst used	Ni/Al ₂ O ₃ (40%)	Ru/TiO₂	Ru/TiO₂	Ru@DOWEX (0,68%)
Catalyst load [g]	0,1	0,06	0,06	0,06
Solvent	Dioxane	Water	Dioxane	Water
Reactor volume [mL]	100	100	50	25
Stirring rate [rpm]	1000	2000	1250	150
Temperature [ºC]	180	90	150	70
H ₂ Pressure [bar]	30	45	45	5
LA concentration [M]	0,22	0,63	0,64	0,43
LA feed [g]	1g	2,9g	2,2g	
S/C molar ratio	-	4350	3200	420
Time [h]	2	6	4	7
LA conversion [%]	100	100	≈ 100	100
GVL selectivity [%]	99,2	89	100	<99

Table 9 Batch reactor experiments

	(Piskun et al., 2016)	(Serrano-Ruiz et al., 2010)	•	arrodan and o, 2014)
Catalyst used	Ru/C (5wt%)	Ru/C (5wt%)	Ru@DOWE	X (0,68wt%)
Catalyst load [g]	2	-	0,0	401
Solvent	Water	Water	Wa	iter
Reactor	Packed bed	Packed bed	Packed bed	concurrent
Temperature [ºC]	90	150	70	70
Pressure [bar]	45	35	5	5,3
LA concentration [M]	1,1	4,6	0,02	0,02
LA feed [mL/min]	1	-	0,15	0,15
H ₂ feed [mL/min]	30		1	1,5
WHSV [h ⁻¹]	30	4,8	-	-
Time [h]	6	-	-	-
LA conversion [%]	92	90	95	100
GVL selectivity [%]	78	96	≈ 100	≈ 100

Table 10 Experiments under continuous flow

Moreno and Marrodan used a bifunctional metal/acid heterogeneous catalyst as said before and by their experiment, we can conclude that using this type of catalyst for the hydrogenation of LA into GVL provides many advantages like for example the metal load compared to other catalyst types can be lower. Moreover, the reaction can be performed with almost 100% yield under mild temperature and pressure which means that the energy consumption decreases.

Therefore, we have chosen as a catalyst for this project a bifunctional metal/acid ion-exchange resin doped with palladium, which has been widely used in hydrogenation reactions, but not in the obtention of GVL from LA.

6. CATALYST

The catalyst used in this project was an AMBERYIST CH28 Polymeric Catalyst. It is beadform, macroporous, sulfonic acid palladium-doped resin. It is specifically suitable for the hydrogenation process at mild temperature, particularly, the hydrogenation of acetone to produce methyl isobutyl ketone (MIBK), since by using palladium as a catalyst, the formation of higher condensation products is avoided. The maximum operating temperature permitted is 130°C, so it is important to design the experiment accordingly. Product Data Sheet is attached in **Appendix 2**, but in **Table 11** there are some of its properties.

Surface Area	36m²/g
Total Pore Volume	0,20cc/g
Average Pore Diameter	260Å
Concentration of Acid Sites	4,8eq/kg
Water Retention Capacity	52-58%
Palladium Load in Dry basis	0,70%
Particle Diameter	850-1050µm
Density	790g/L
Maximum Operating Temperature	403K
LHSV Suggested	0,5-5h ⁻¹

Table 11 AMBERLYST CH28 properties (Dupont de Nemours)

6.1. ACTIVATION

This type of catalyst needs to be activated before being used for the reaction desired. Activate the catalyst means to reduce the catalytic precursor to its metallic form. There are several papers about the use of AMBERLYST CH28 in the synthesis of MIBK from acetone, but only a few about the hydrogenation of LA into GVL. The activation procedure selected would be based on MIBK reaction works, because they are explained with more detail, and it is expected that the catalyst

activation is effective, in a similar way, for both reactions, considering that experimental conditions will be also very similar.

Jose A. Trejo et al activated the catalyst in a packed bed reactor where the reaction took place, making circulate hydrogen at 100°C and 20bar (Trejo et al., 2010). Whereas W. Nicol and E. L. du Toit didn't reduce the palladium previously. The catalyst was activated by allowing acetone (3,3mL/min) and hydrogen (330mL/min), which are the reactants of the reaction that would be catalysed, at 30bar flow through the catalyst bed for 4h. Then, reaction conditions were implemented, and experiments went on. It was checked that after 4h no more reduction of Pd took place, so the catalyst could be considered activated (Nicol and Du Toit, 2004). Both of them activated AMBERLYST CH28 before performing the obtention of MIBK from acetone.

It has been reported that the catalyst deactivation is caused by polar compounds like water, due to their strong affinity towards acid catalytic sites. When water molecules are in a non-polar environment, they absorb on the resin surface and cover the catalytic sites inhibiting the sulfonic acid groups of the catalyst. Talwalkar and Mahajani tested the activity of the AMBERLYST CH28 in the reaction of MIBK production from acetone and found that the deactivation is reversible. Once the catalyst had been used, it was separated and dried in an oven for 3h and then re-used; the performance was almost the same (Talwalkar and Mahajani, 2006). It is important to point out that the deactivation caused by water is relatively slow. For the hydrogenation of LA into GVL, another possible factor that leads to the deactivation of the catalyst tolerate (130°C) is much lower than the temperatures that lead to AL formation (300-350°C), so it won't be a problem for us.

The fabricant recommends reducing the Pd making circulate H_2 and deionised water for 16h through the catalytic bed. The H_2 will reduce the Pd and the water will solubilise the H_2 and swell the catalyst.

7. EXPERIMENTAL DESIGN

The objectives of this project were at the beginning making a kinetic study and found the optimal conditions to maximise the production of GVL from LA using AMBERLYST CH28. We prepared and proved the equipment. We did some experiments to have an idea about in which conditions the reaction should be, but when we were going to start with the kinetic study experiments, the laboratories of the University were closed due to the pandemic, so we couldn't carry out the experiments.

7.1. FACILITIES

The equipment available to be used was the Microactivity Reactor and a gas chromatograph. Both are controlled by a computer equipped with the corresponding software. In addition, Microactivity Reactor has a touch screen from where it can be controlled. In **Figure 16**, the facility is shown. On the left side, Microactivity Reactor, in the middle there is the chromatograph and, the computer is on the right side.



Figure 16 Equipment

Microactivity Reactor consists of a tubular fixed bed reactor, where the catalyst is set inside, upon a porous plat (**Figure 17**). The flow was thought to be up to down, but in our case, the flow will be ascendant. The reasons will be explained later. The gas reactants are feed to the reactor passing through a shut-off valve and measured by a mass flow controller. Liquid reactants are dosed by HPLC alternative positive displacement pump located over the Microactivity Reactor (streams ranging 0,01-5mL/min, and pressure up to 600bar), and introduced into the system through a low dead check-vale. Liquid and gas are connected inside the Hot Box in a 3-port valve.



Figure 17 Reactor and porous plate

The Hot Box (**Figure 18**) has several devices inside: the tubular reactor with an electric heating jacket, an electric forced convection heater (to keep the Hot Box at temperatures up to 180°C), a 6-port valve, a pressure regulator, two temperature sensors, one in the reactor and another in the Hot Box, and a pressure sensor.



Figure 18 Hot Box

Once the liquid and the gas streams have been mixed in the 3-port valve, they go through the 6-port valve, where depending on which configurations have been set on the computer, leads the flow to the reactor or directly out of the hot box (by-passing the reactor). The flow passes through the reactor and then it returned into the 6-port valve again to go out of the Hot Box into the liquidgas separator deposit located at the back of the Microactivity Reactor (**Figure 19**, the deposit is located at the bottom, at the caved area). This element is thought to cool the products mixture (by a Peltier cell) to condensate and separate the liquids from the gas. Since it has a calibrated level sensor, it will also provide the liquid level of the deposit in real-time. From the computer, it is possible to set the liquid level desired by opening or closing a valve located at the liquid outlet. As the gases of our reaction are H_2 and N_2 (it will be used even it is not involved in the reaction), and H_2 is dangerous, they will be led by a tube into a gas extractor after being returned into the Hot Box to measure the pressure and regulate the opening of a servo valve to maintain the pressure at the desired value. The liquid current is sent to the gas chromatograph, where vaporised and injected to be analysed. The PI&D and some other layouts are attached in **Appendix 1**.



Figure 19 Back of Microactivity-Reactor

All the parameters can be configurated from the computer and some of them from the touch screen too. The Microactivity Reactor also has 4 displays in which are shown the setpoint and the value of the Hot Box temperature, the reactor temperature, the pressure, and the liquid level of the deposit. In the front of the Microactivity Reactor, there are also two manual valves to regulate the gas flow, one for H_2 and one for N_2 . Although they can be partially open, we will use them always completely open or closed and we will regulate the flow from the computer or the touch screen.

The chromatograph is connected to the system, so the samples are injected just by ordering it from the computer, but also, samples can be injected manually. The method used can be seen in **Appendix 3** (Model 2). At the laboratory, also a mass spectrometer is available, but it was being used, so we just used it for punctual analysis to compare results from our chromatograph.

The liquid reactants are settled over the equipment in bottles connected to the pump as can be seen in **Figure 16**. Gas reactants are in gas bottles. The N_2 is in the laboratory and the H_2 is in the roof of the Faculty, in a properly conditioned room.

7.2. SUBSTANCES

Acetone: we used to clean the needle and to calibrate the chromatograph.

 α -angelica lactone: reaction intermediate. It is used to calibrate the chromatograph and by comparison, see if it is formed or not. It is not dangerous, and it is bough to Sigma Aldrich. Its purity is 98%.

AMBERLYST CH28: catalyst. It has already been talked about before (chapter 6). It is gently supplied by Dupont.

Deionized water: solvent to prepare the aqueous solution of LA that will feed the reactor. It is deionised tap water.

y-valerolactone: product. It has already been talked about before (chapter 4). It is not hazardous. It is bough to Sigma Aldrich. Its purity is 99%.

Hydrogen: reactant. It comes to the reactor from the high-pressure line. It is important to be aware that it is very inflammable. It is high purity hydrogen 5.0 (more than 99,999%) bought in a high-pressure bottle to Abelló Linde.

Levulinic Acid: raw material, one of the reactants. It is irritant, so it must be treated carefully. As said before (chapter 3), it is solid at room temperature, but easily turned into liquid. It is slightly yellowy. Its purity is higher than 98%. It is bought to Acros Organics.

Nitrogen: it is used to adjust the pressure and to test the system to assure that there are no escapes before opening the hydrogen valve due to it is not dangerous and it doesn't react with the other substances. It is also high purity nitrogen 5.0 (more than 99,999%) bought in a high-pressure bottle to Abelló Linde.

Silicon Carbide powder, coarse, 46 grit: inert to mix with the catalyst. It is a carcinogen, so it should be treated with gloves and with a face mask. It is bought to Alfa Aesar.

Valeric Acid (Pentanoic Acid): possible product. Its purity is higher than 99%. It was bought to Sigma Aldrich.

4-hydroxypentanoic acid: intermediate. It is not available, so to be detected, the mass spectrometer must be used.

More information about the substances is attached in Appendix 2.

7.3. **EXPERIMENTAL**

7.3.1. Equipment set up

The first thing we did was preparing the equipment. As mentioned before, Microactivity Reactor had been used in other processes and the installation was not suitable for our reaction, so some changes were done. The flow direction was thought to be a descendant, to avoid the fluidization of the bed. But some problems can appear with this configuration such as compaction of the bed which involves extremely high-pressure drop and the segregation of bed particles which means the creation of regions without the catalyst. Since our flow velocities are considerably low the fluidization won't be possible, therefore we decided that the stream should ascend through the bed, improving the contact.

7.3.2. Determinate retention times

Then before starting to operate with the Microactivity Reactor, we prepare some samples with liquid compounds involved in our reaction to analyse them at the chromatograph. This analysis would be useful to determine the retention time of the substances. Every sample (1mL) had the same composition (1mL GVL, 1mL LA, 1mL valeric acid, and 5mL H₂0) and we added 2 mL of one of them to every sample and injected 1mL of the solution resultant to the chromatograph. Chromatograms show an area for each substance and that area is proportional to the mass of that compound in the mixture. So, since we had one substance in a higher proportion than the others, it would be represented with a bigger area, and the retention time of the substance would be determined. Apart from adding the reaction substances, we also added acetone to some samples, because as we clean the needle with acetone, some can be detected when we inject samples manually to the chromatograph. We included valeric acid because it was possible to obtain it if we continue to the obtention of 5-nonanol. We also analyse a sample with AL in order to can detect it in case it was formed. The results of these chromatograms are attached in **Annex 4** (chromatograms 1-7), and the retention time of each substance is in **Table 12**.

	Retention time [min]
H ₂ 0	3,70
Acetone	4,51
AL	7,89
Valeric Acid	8,31
GVL	8,56
LA	9,34

Table 12 Retention time	s
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7.3.3. Feed solution

We also prepared the aqueous solution of LA that would be fed to the reactor. We prepared 500mL of a 50%v/v solution (250mL deionised water + 250mL LA). With 500mL we could perform more than one experiment. We needed to heat the LA bottle in a heated bath to turn it liquid.

7.3.4. Test SiC reactivity

The reactor was loaded with a mixture of catalyst and SiC. Introducing SiC particles, with a smaller diameter increased the pressure drop, but it helps to approach the flow to a plug flow reactor and makes the bed more isothermal. Since the catalyst is expensive, even the energy requirements are quite bigger due to the increase in the pressure drop, the reduction of the amount of catalyst makes this decision worthy. SiC is also expensive, but it is inert, so it can be recovered and use it for more than one experiment. To be sure that SiC is inert and does not catalyse the reaction, we loaded the reactor with it (5mL) and operate (0,5mL/min LA 50%v/v, 100mL/min H₂, 210°C, 15bar). We injected a sample into the chromatograph after a few hours of operation and no product was detected(**Appendix 4**, chromatogram 8), so as it was expected, SiC is inert.

7.3.5. Catalyst preparation

Before filling the reactor with the catalyst, it must be washed with deionised water to remove the powder (the tiny particles would increase the pressure drop of the bed). Then we dried the catalyst, first letting it in a piece of filter paper and then in an oven for some hours. It is important to eliminate all the moisture as possible from the catalyst, because when the water with hydrogen goes through the bed during the activation, if the catalyst has already water in it, the hydrogen won't arrive at all the Pd. During this step, we measure the volume and weight of the dry and wet catalyst to determine the density and the expansion accurately. The results are in **Table 13**.

	Dry	Wet
Weight [g]	2,74	5,42
Volume [mL]	3,5	7,5
Density [g/mL]	0,78	0,72

Table 13 Dry and wet catalyst properties

We took a sample of the catalyst to an external laboratory to know more about it, but when we had to stop because of the university closure, the results hadn't arrived.

7.3.6. Trial Experiments

Before planning the conditions to study the reaction, we had been doing some experiments to get familiarised with the equipment and to know if the reaction took place in different conditions. At first, we didn't know the amount of catalyst to load. Finally, we found that (as will be explained later) with 1g of catalyst and 0,5mL of SiC everything worked so we continued with that load volume. We started feeding the reactor with 0,5mL/min of H₂O and 30mL/min of N₂ (to ensure the equipment as will be explained later) when the control was working properly, we started to feed 10mL/min of H₂. In the beginning, we couldn't feed the reactor with more than 10mL/min of H₂, so we had to continue with the N₂ feed to keep the pressure value. We replace the water stream by 0,2mL/min of LA solution at 130°C and 20bar and after a few hours, we started to inject samples to the chromatograph. Then we fixed the problem with the H₂ feed, so we repeated the experiment increasing the H₂ flow from 10mL/min to 150mL/min and without keeping the N₂ feed.

7.3.7. Experiments

Each experiment is thought to be developed during a day, in about the 10h that the laboratory is available because the equipment must be stopped during the night and started the next day. Hence the times had to be adapted and all the steps should be well planned and carried out nimbly.

Load the reactor: The reactor is a tubular reactor of about 5mL. According to the catalyst expansion that we had determined when we prepared it (**Table 13**), we decided that the bed would be formed by 1g of dry catalyst (1,27mL of the dry catalyst which means about 2,7mL of wet catalyst) and 0,5mL of SiC. The total volume of the bed once the catalyst has retained the water is 3,2mL approximately. It is less than the volume of the bed, but we did some trials and realised that if we filled the bed with more volume of catalyst or SiC, the pressure drop was too high, and the bed expanded too much, so the system collapsed. It must be loaded from the upper hole due to in the lower part, the porous plate is placed to support the bed.

<u>Set the reactor</u>: Once the reactor is loaded, it has to be assembled into the Hot Box. It is hard work because all the joints must be well tightened to avoid leaks.

Ensure the equipment: When the reactor is placed, Microactivity Reactor can be switched on. Firstly, we open the H_20 and N_2 valves (0,5mL/min H_2O and 100mL/min N_2) to reach a pressure of 30bar and drag possible rests from previous runs. We check all the joints to be sure that there are no leaks. While this test is being done, we also set the parameters of control (**Table 14**) according to the Microactivity Reactor user's manual and let time to the controllers stabilise the controlled parameters near to setpoint values. Once the controllers are regulating the pressure, temperatures (reactor and Hot Box), and liquid level, and everything is with no leaks, we change the water feed to LA solution and go through the next step.

	Liquid Level	Pressure	T. Hot Box	T. Reactor
Set point	4	20	120	110
Р	200	200	15	60
I	15	15	350	400
D	0	0	50	50
RP1	0	0	0	0
MH1	70	90	95	30
ML1	0	0	0	0
SLH	10	100	200	120
SLL	2,5	0	0	0

Table 14 Control parameters

<u>Catalyst activation</u>: Although the fabricant made a recommendation to activate AMBERLYST CH28, we couldn't afford the 16h of activation that this process involves. We didn't know if using

water and H₂ as recommended, for a shorter time the catalyst would be active. We didn't know flow rates values either. Therefore, we decided to follow the activation performed by Nicol and Du Troit because they found that in 4h it was activated, and that time was suitable for our experiment. However, we had to adjust some of their conditions. They activated AMBERLYST CH28 to synthesize MIBK in 2004 (Nicol and Du Toit, 2004). Their reactor was considerably bigger, so they used more catalyst (49,5mL of wet catalyst) than us. Our reactor is smaller, but we decided to conserve approximately the same LHSV that they used (4h-1). Considering that we load our reactor with 1g of catalyst, the feed flow needed is 0,18mL/min. As mentioned before (Chapter 6), they used the reaction reactants to activate the catalyst, in our reaction, they are LA 50% v/vand H₂. About gas flow, since we consider that 1/10 of the H₂ is absorbed, the molar flow should be 10 times bigger to be sure that there is enough to react. Hence, the H₂ feed required is 14mL/min. We had problems with the pressure, we needed to use at least 50mL/min so we aren't able to conserve the ratio gas flow/liguid flow (they used 100 and our is about 277) and even though, we couldn't reach 30bar, we operate at 20bar. A possible solution to conserve the ratio and pressure is feeding the reactor with the H_2 needed and add N_2 until the pressure is 30bar (14mL/min H₂ + 50 mL/min N₂), but we didn't try it. We use the same temperature 130°C since it is the maximum operating temperature allowed by the catalyst. To sum up, all the conditions are tabulated in Table 15.

Feed stream	Aqueous solution LA 50%v/v + H ₂
Catalyst dry weight (g)	1
Wet Catalyst volume (mL)	2,74
Volume of solid inert (mL)	0,5
LHSV (mL feed/(mL cat·h))	3,95
Liquid volumetric flow rate (mL/min)	0,18
Gas volumetric flow rate (mL/min)	50mL H ₂ or 14mL H ₂ + 50mL N ₂
Temperature (°C)	130
Pressure (bar)	20bar or add N ₂ until 30bar
Activation time (h)	4

Table 15 Catalyst activation

<u>Reaction</u>: The time of this step would be about 4 hours. If we could have done everything as we expected before the pandemic, we would have designed different experiments with different reaction conditions to analyse the results and do the kinetic study and if we had time, the

production study. To perform the kinetic study, the first thing to do is study the reaction rate varying the flow velocity to determine the dependence between the flow velocity and the mass external transfer (MET). Then with the flow velocity fixed, the particle size will be varied to find the dependence between the particle diameter and the mass internal transfer (MIT). Once a diameter and a flow velocity are determined to be sure that MIT and MET don't control, we have to carry out different runs with different LA and H₂ concentrations at different temperatures until the steady-state is reached. We will work in a differential regime to obtain reaction rates at different temperatures and adjust a kinetic model.

<u>Stop</u>: To stop the reaction system, the H₂ is replaced by N₂ and the LA solution by water to drag all the fluid that rest in the system. The valve which controls the liquid level of the deposit must be opened. After a few minutes, the feed can be closed, and Microactivity Reactor switched off.

<u>Clean</u>: The reactor is removed from the Hot Box and emptied washing it with water recovering and separating the catalyst and the inert. The inert can be used in another run and once the deactivation of the catalyst is tested, we have to decide if it can be prepared again to be used in another run or not.

<u>Notes</u>: As has been mentioned, assembling and removing the reactor is hard and takes time, so to take more profit of the day, it is better to once the experiment of the day is finished, clean and prepare the facility so that the next day the first step can be ensure the equipment.

7.3.8. Results

We couldn't carry out the important experiments because of the pandemic, but we could do some trials where GVL was detected by the gas chromatograph. The first time we detected GVL was when we used 10mL/min of H₂ (the first trial experiment explained). Even though, the peaks of the chromatogram were superposed, so it was not clear. This chromatogram can be seen in **Appendix 4** (chromatogram 9). With the second experiment (150mL/min H₂) the peak was clear. **Figure 20** corresponds to this last chromatogram, and by comparing the retention times, we can conclude that GVL had been produced. We can't know the amount produced, because we didn't have time to calibrate the chromatograph to associate areas with volume or mass composition, but the area was enough to suppose that GVL was in a good proportion. We also can determine,

that by the time we analysed the sample, any by-product was formed, so selectivity to GVL is 100%. This chromatogram is attached in **Appendix 4** (chromatogram 10).



Figure 20 Chromatogram

8. CONCLUSIONS

The exhaustion of petroleum has created the necessity to research to find alternatives to end up with petroleum dependence. Moreover, the environmental problems associated to its derived products must be fixed so the aim is to find renewable sources able to replace oil as a raw material. Petroleum consumption is stabilised in almost all the sectors but not in transportation, where it is still increasing. Since transportation is where the most amount of oil is used, it is important to find alternatives. Electric cars have been proposed since a long time ago, but there is much research to be done before they can displace gasoline and diesel cars. Biomass is another alternative that is gaining more importance due to its abundance in the world and its renewable nature. From biomass, many petrochemical products can be replaced with similar properties and more advantages such as fewer environmental hazards. However, biomass can only partially replace petroleum uses with the technology available.

GVL is lignocellulosic biomass-derived that can be produced from LA, which is considered one of the most promising products due to its wide application areas as a product or as an intermediate to other products. The fact that lignocellulose comes from non-edible biomass avoids the ethical problem posed by using food crop plants in areas not related to alimentation.

At first, GVL was not suitable to be produced at industrial scale because it was too expensive to be produced, but while LA production technologies are improving (Biofine process is the closest to be industrialised), GVL production costs are considerably lower, so its commercialization is becoming feasible at a competitive price compared to petroleum-derived products. The principal uses of GVL are related to fuels: as a biofuel, as a precursor to other hydrocarbon fuels, or as an additive. Moreover, it is suitable to produce other petrochemical derivates such as polymers or use it as a solvent to several biomass derivates production processes.

The reaction to produce GVL from LA has two possible pathways. The first and desired consists of hydrogenation catalysed by a metal catalyst followed by internal esterification catalysed by an acid catalyst. The second pathway involves dehydration of LA to produce AL and the subsequent hydrogenation to obtain GVL. AL causes problems such as lower GVL yields and

depositions on the acid sites of the catalyst avoiding its good performance. But with mild operation conditions, AL formation doesn't take place, so the desired pathway is followed.

We operate in a tubular reactor of about 5mL loaded with 1g of dry catalyst and 0,5mL of SiC (inert). We used a bifunctional catalyst loaded with Pd (AMBERLYST CH28) which needs to be reduced before the reaction. We made the activation at 130°C, which is the maximum temperature the catalyst can tolerate, using an aqueous solution of LA 50%v/v at 0,18mL/min and 50mL/min of H₂ at 20bar for 4h. We couldn't carry out the kinetic and production studies, but we did some runs and operating with 0,2mL/min of the LA solution and 150mL/min H₂ at 130°C and 20bar, after 2h of reaction, we found that GVL is formed in good selectivity.

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ACRONYMS

AL:		α-angelica lactone
DA	LA:	δ-aminolevulinic acid
DP	A:	Diphenolic acid
GV	L:	γ-valerolactone
HM	IF:	Hydroxymethylfurfural
LA:		Levulinic acid
LH	SV:	Liquid hourly space velocity, ratio of liquid volume flow per hour (mL/h) to catalyst
volume (mL)		
ME	T:	Mass external transfer
Me	THF:	Methyltetrahydrofuran
MIE	BK:	4-methyl-2-pentanone / methyl isobutyl ketone
MIT	Г:	Mass internal transfer
WH	ISV:	Weight hourly space velocity, ratio of weight feed flow per hour (g/h) to catalyst
weight (g)		

APPENDICES

APPENDIX 1: MICROACTIVITY REACTOR PI&D








APPENDIX 2: INFORMATION ABOUT SUBSTANCES

Description AA aci Th (M pa co vpplications • 'ypical Properties P	IUSTRIAL-grade, Palladium-do MBERLYST™ CH28 Polyr Id, palladium-doped resin is catalyst is especially su IBK) from acetone. In this Iladium that is loaded on t Iladium that is loaded on t Iladium that is loaded on t Hydrogenation (MIBK) hysical Properties Copolymer Matrix Type Functional Group Physical Form Itrogen BET Surface Area	R8 Polymeric Catalyst pped, Strongly Acidic Catalyst meric Catalyst is a bead-form, macroporous, sulfonic developed particularly for heterogeneous catalysis. iitable for the production of methy isobutyl ketone case, the hydrogenation reaction, catalyzed by the he resin, prevents the formation of higher Styrene-divinylbenzene Macroporous Strong acid cation Sulfonic acid Gray, opaque, spherical beads
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	Matrix Type Functional Group Physical Form litrogen BET Surface Area	Macroporous Strong acid cation Sulfonic acid
	Type Functional Group Physical Form itrogen BET Surface Area	Strong acid cation Sulfonic acid
	Functional Group Physical Form itrogen BET Surface Area	Sulfonic acid
	Physical Form itrogen BET Surface Area	
	Surface Area	Gray, opaque, spherical beads
	Surface Area	
c		
C		36 m²/g
c	Total Pore Volume	0.20 cc/g
c	Average Pore Diameter	260 A
	hemical Properties	
	Ionic Form as Shipped	H ⁺
	Concentration of Acid Sites ‡	≥ 4.80 eq/kg
		≥ 1.60 eq/L
	Water Retention Capacity	52 - 58%
	Palladium Load	
	Dry basis	≥ 0.70%
_	Wet basis article Size [§]	≥ 2.4 g/L
P		050 4050
	Particle Diameter Uniformity Coefficient	850 – 1050 μm ≤ 1.40
	< 710 µm	
	> 1180 µm	≤ 2.0% ≤ 15.0%
	hrinkage (in solvent)	- 10.074
5	Acetone	14%
	MIBK	19%
	ensity	1070
5	Shipping Weight	790 g/L

F Ury Weight Capacity ≥ 4.80 eq/kg; Total Exchange Capacity (on a water wet basis) ≥ 1.60 eq/L 9 For additional particle size information, please refer to the <u>Particle Size Distribution Cross Reference Chart</u> (Form No. 177-01775).

Form No. 177-03100, Rev. 2 June 2019

Suggested Operating Conditions

130°C (265°F)	
1000 mm (3.3 ft)	
1 bar (15 psig) across the bed	
0.5 - 5 h ⁻¹	
See Figure 1	
	1000 mm (3.3 ft) 1 bar (15 psig) across the bed $0.5 - 5 h^{-1}$

Hydraulic Characteristics

Estimated bed expansion of AMBERLYST™ CH28 Polymeric Catalyst as a function of backwash flowrate and temperature is shown in Figure 1.

Estimated pressure drop for AMBERLYST[™] CH28 as a function of service flowrate and temperature is shown in Figure 2. These pressure drop expectations are valid at the start of the service run with clean water and a well-classified bed.



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	Please be aware of the following: • WARNING: Oxidizing agents such as nitric acid attack organic ion exchange resins under certain conditions. This could lead to anything from slight resin

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22/6/2020	ICSC 0087 - ACETONE
ACETONE	ICSC: 0087
2-Propanone	
Dimethyl ketone	April 2009
Methyl ketone	
CAS #: 67-64-1	
UN #: 1090	
EC Number: 200-662-2	

Highly flammable. Vapourlair mixtures are explosive. Heating will EXPLOSION cause free in pressure with risk of burstino.	NO asses fismes AlO assesses and NO	
	models grant we save a supervise the second seco	Use powder, alcohol-resistant foam, water, carbon dioxido. In case of fire: keep drums, etc., cool by spraying with water.

	SYMPTOMS	PREVENTION	FIRST AID
Inhalation	Sore throat. Cough. Confusion. Headache. Dizziness. Drowsiness. Unconsciousness.	Use ventilation, local exhaust or breathing protection.	Fresh air, rest. Refer for medical attention.
Skin	Dry skin.	Protective gloves.	Remove contaminated clothes. Rinse skin with plenty of water or shower.
Eyes	Redness. Pain. Blurred vision.	Wear safety spectacles.	Rinse with plenty of water (remove contact lenses if easily possible). Refer for medical attention.
Ingestion	Nausea. Vomiting. Further see Inhalation.	Do not eat, drink, or smoke during work. Wash hands before eating.	Rinse mouth. Refer for medical attention .

i indiana iRinan alana animi inana inina	CLASSIFICATION & LABELLING	According to UN CH3 Criteria	DANGER	Highly flammable liquid and vapour Causes eye irritation	Transportation UN Classification UN Hazard Class: 3; UN Pack Group: II	or behalf of ILO and WHO.
	SPILLAGE DISPOSAL	Remove all piloton sources are forwarding point adapted to the regarding to un child Criteria recognic games and vigours of forwarding point adapted to the forecal methore concentration of the adamters. Winterform Coderd and or free adapted and the adapted of the adapted of the local regulations. Do NOT watch and virtua severe local regulations. Do NOT watch and virtua severe	STORAGE	Fireproof. Separated from : see Chemical Dangers. Store in an area without drain or sewer access.	PACKAGING	Come of the second seco

ACETONE	ICSC: 0087
PHYSICAL & CHEMI	PHYSICAL & CHEMICAL INFORMATION
Physical State: Apparance COLOURLESS LIQUID WITH CHARACTERISTIC ODOUR. Physical dangers	Formula: C.J.N.O. C.H.3-CO-C.H.3 Molecular mass: 58.1 Melling point: 65°C
The vapour is heavier than air and may travel along the ground; distant ignition possible.	Relative density (water = 1): 0.8 Solubility in water: miscible Vapour pressure, kPa at 20°C: 24
contract analysis of the second and contract and and contract who should not contract and second and contract who should be added with the contract of the second second second and contract of the second second second second and explosion hazard. Alacks pleaks, and explosion hazard. Alacks pleaks,	The second method is a (* 1) 2.0 Key the second method is a first of the support of the support is a first of the support of t
EXPOSURE & HE	EXPOSURE & HEALTH EFFECTS
Receive of exposure The culture of the accord rise the lookly by inhalation. These at provident exposure Effects of another reports a high freets could stand browing of consolutions.	Measurements Measurements expositions of the action be reached rather quickly on expositions of these substances at 20°C, on spraying or dispersing much items factor at interpret on a system exposition control of the factor at interpret or a system exposition control of the factor at interpret or a system exposition control of the factor at interpret or a system exposition control of the factor at interpret or a system exposition control of the factor at interpret or a system exposition control of the factor at interpret or a system exposition control of the factor at interpret or a system exposition control of the factor at interpret or a system exposition control of the factor at interpret or a system exposition control of the factor at interpret or a system exposition control of the factor at interpret or a system exposition control of the factor at interpret or a system exposition control of the factor at interpret or a system exposition control of the factor at interpret or a system exposition control of the factor at interpret or a system exposition control of the factor at interpret or a system exposition control of the factor at interpret or a system exposition control of the at interpret or a system exposition control of the factor at interpret or a system exposition control of the at interpret or a system exposition control of the factor at interpret or a system exposition control of the at interpret or a system exposition control of the factor at interpret or a system exposition control of the at interpret or a system exposition control of the factor at interpret or a system exposition control of the at interpret or a system exposition control of the at interpret or a system exposition control of the at interpret or a system exposition control of the at interpret or a system exposition control of the at interpret or a system exposition control of the at interpret or a system exposition control of the at interpret or a system exposition control of the at interpret or a system
OCCUPATIONAL E	OCCUPATIONAL EXPOSURE LIMITS
TLV: 520 ppm as TWX; 500 ppm as STEL; BEI issued; A4 (not dassifiable as a human carcinogen). AMX: 1200 mg/m ³ 500 ppm ; peak limitation category: I(2); pregnancy risk group: B. EU-OEL: 1210 mg/m ³ 500 ppm as TWA	sssifiable as a human carcinogen). ancy risk group: B.
ENVIRO	ENVIRONMENT

NOTES	Use of alcoholic beverages enhances the harmful effect.	ADDITIONAL INFORMATION	EC Classification Symbol: F, Xi; R: 11-36-65-67; S; (2)-9-16-26
	Use of alcoholic be		EC Classification Symbol: F, Xi; R: 114

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file:///H/JUNI/TFG/TFG EVA/Is insst/ICSC 0087 - ACETONE.html

www.sigmaaldrich.com	
Sigma-Aldrich.	

FICHA DE DATOS DE SEGURIDAD de acuerdo el Reglamento (CE) No. 1907/2006

Versión 6.0 Fecha de revisión 21.04.2019 Fecha de impresión 13.06.2020 SECCIÓN 1. Identificación de la sustancia o la mezcla y de la sociedad o la empresa

1.1 Identificadores del producto Nombre del producto

a-Angélicalactona

Referencia	 A86406
Marca	 Aldrich
REACH No.	 Un número de registro no está disponible para
	ya que la sustancia o sus usos están exentos o
	tonelaje anual no requiere registro o dicho reg

a esta sustancia,

del registro, el gistro está previsto para una fecha posterior 591-12-8

Usos pertinentes identificados de la sustancia o de la mezcla y usos desaconsejados 1.2

No. CAS

: Reactivos para laboratorio, Fabricación de sustancias Usos identificados

Datos del proveedor de la ficha de datos de seguridad 1.3

: Merck Life Science S.L. Calle Maria de Molina 40 Compañía

Teléfono : +34 916 619 977 Fax : +34 916 619 642 Fax : +34 916 619 642 E-mail de contacto : serviciotecnico@merckgro		Ш	E-28006 MADRID
	Teléfono	с+ 	14 916 619 977
	Fax	÷	14 916 619 642
	E-mail de contacto	: se	rviciotecnico@merckgrou

up.com

1.4 Teléfono de emergencia

: 900-868538 (CHEMTREC España) +(34)-931768545 (CHEMTREC nternacional) Teléfono de Urgencia

SECCIÓN 2. Identificación de los peligros

2.1 Clasificación de la sustancia o de la mezcla

- No es una sustancia o mezcla peligrosa de acuerdo con el Reglamento (CE) No. 1272/2008.
- 2.2 Elementos de la etiqueta
- No es una sustancia o mezcla peligrosa de acuerdo con el Reglamento (CE) No. 1272/2008. Otros Peligros - ninguno(a) 2.3

SECCIÓN 3. Composición/información sobre los componentes

3.1 Sustancias

Aldrich- A86406

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: 5-Methyl-2(3H)-furanone	4-Hydroxy-3-pentenoic acid y-lactone	alpha-Angelica lactone	
Sinónimos			

: C5H6O2	: 98,10 g/mol	: 591-12-8	: 209-701-8
Formula	Peso molecular	No. CAS	No. CE

α-ANGELICA LACTONE

Según la normativa aplicable no es necesario divulgar ninguno de los componentes.

SECCIÓN 4. Primeros auxilios

Descripción de los primeros auxilios 4.1

Recomendaciones generales

Consultar a un médico. Mostrar esta ficha de seguridad al doctor que esté de servicio. Si es inhalado

Si aspiró, mueva la persona al aire fresco. Si ha parado de respirar, hacer la respiración artificial. Consultar a un médico.

En caso de contacto con la piel Eliminar lavando con jabón y mucha agua. Consultar a un médico.

Lavarse abundantemente los ojos con agua como medida de precaución. En caso de contacto con los ojos

Por ingestión

No provocar el vómito. Nunca debe administrarse nada por la boca a una persona inconsciente. Enjuague la boca con agua. Consultar a un médico.

Principales sintomas y efectos, agudos y retardados Los síntomas y tectos más importantes conocidos se describen en la etiqueta (ver sección 2.2) y / o en la sección II. 4.2

Indicación de toda atención médica y de los tratamientos especiales que deban 4.3

dispensarse inmediatamente Sin datos disponibles

SECCIÓN 5. Medidas de lucha contra incendios

5.1 Medios de extinción

Medios de extinción apropiados

Usar agua pulverizada, espuma resistente al alcohol, polvo seco o dióxido de carbono. Peligros específicos derivados de la sustancia o la mezcla 5.2

- Óxidos de carbono
- Recomendaciones para el personal de lucha contra incendios Si es necesario, usar equipo de respiración autónomo para la lucha contra el fuego. 5.3
- Otros datos 4.5
- El agua pulverizada puede ser utilizada para enfriar los contenedores cerrados.

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		Drotección de la niel		
rocedimientos de emergencia los vapores, la neblina o el gas. los vapores que se acumulan ueden acumularse en las zonas		Manipular con guant Utilice la técnica conr guante) para evitar e contaminados despu prácticas de laborato	revocation at previous previous a previous deben ser inspectionados antes de su uso. Heamplate con guantes: Los guantes los guantes (sa fucar a superindre exterior del guante) para evitar el cunacto de la pile con este producto. Descrite los guantes contaminados de la pile con este producto. Descrite los guantes prácticas de laboración. Lavar y secer las manos.	iso. erior del antes buenas
		Los guantes de prote del Reglamento (UE)	Los guantes de protección seleccionados deben de cumplir con las especificaciones del Reglamento (UE) 2016/425 y de la norma EN 374 derivada del mismo.	aciones
n riesgos. No dejar que el		Protección Corporal Indumentaria imperme según la concentraciór trabajo.	Protección Corporal Indumentaria impermeable. El tipo de equipamiento de protección debe ser elegido apoli e concentración y la cantidad de sustancia peligrosa al lugar específico de trabajo.	elegido co de
in las la ectricidado o cepilandolo, y na las reglamentaciones locales r cerrados para su eliminación.		Protección respiratoria Donde el asesoramiento o cara con combinacion mu el respirador es la unica p componentetes testados y apropiados como NIOSH (Producción trajetitadoria Donde di assessamiento de respo muestre que los respiradores purificadore toda la carea con compateixa (en un posto de la funciona de la grandare en asi el respiradore si la unicio producción, usar un respiradore S usar respiradores y componentes estandos a polito de cabilito de la defensa estándarda publicadores y propuedas como NOSH (EEUU) o CEN (UE)	toda la eria. Si e
		Control de exposición ambiental Impedir nuevos escapes o derrames producto entre en el sistema de alca	control de exposición ambiental Impedir nuros escapes o derrames i puede hacerse sin riesgos. No dejar que el producto entre en el isstema de aciantanilado.	que el
o fumar.Tomar medidas para				
	SECCIÓ	SECCIÓN 9. Propiedades físicas y químicas	tas y químicas	
posibles incompatibilidades	9.1 In	formación sobre prop	9.1 Información sobre propiedades físicas y químicas básicas	
héticamente cerrado en un lugar eben volverse a cerrar	a)	a) Aspecto	Forma: claro, líquido Color: amarillo claro	
rital perunas.	(q	Olor	Sin datos disponibles	
	C)	Umbral olfativo	Sin datos disponibles	
estipulan otros usos específicos	(p	ЬН	Sin datos disponibles	
	(ə	Punto de fusión/ nunto de congelación	Punto/intervalo de fusión: 13 - 17 °C - lit.	
	(J	Punto inicial de	55 - 56 °C a 12 mmHg - lit.	
cposición profesional.		ebuilición e intervalo de ebullición		
i proresional.	(6	Punto de inflamación	68 °C - copa cerrada	
	(u	Tasa de evaporación	Sin datos disponibles	
cuadas, y respetar las prácticas s v desoués de terminar la	(1	Inflamabilidad (sólido, gas)	Sin datos disponibles	
	(í	Inflamabilidad superior/inferior o límites explosivos	Sin datos disponibles	
ormes con la EN166 Use equipo	()	Presión de vapor	Sin datos disponibles	
gún las normas gubernamentales	(1	Densidad de vapor	Sin datos disponibles	
4 166 (UE).	(m		1,092 gcm3 a 25 °C	
	(u	Solubilidad en agua	Sin datos disponibles	
Pagina 3 de 8	Aldrich- A86406	6406		Pagina 4 de 8
Merck	The life so	ience business of Merck o	The life science business of Merck operates as MilliporeSigma in	JERCK
	the US and Canada	a Canada		

SECCIÓN 6. Medidas en caso de vertido accidental

6.1 Precauciones personales, equipo de protección y pro Utilicese equipo de protección individual. Evitar respirar los Retirar todas las fuentes de ignición. Tener cuidado con los formando asi concentraciones explosivas. Los vapores pue Equipo de protección individual, ver sección 8. inferiores.

Precauciones relativas al medio ambiente 6.2

Impedir nuevos escapes o derrames si puede hacerse sin producto entre en el sistema de alcantarillado.

6.3

Métodos y material de contención y de limpieza Content y recoger el derrame con un aspirador aislado de meterlo en un envase para su eliminación de acuerdo con (ver sección 13). Guardar en contenedores apropiados y co

Referencia a otras secciones Para eliminación de desechos ver sección 13. 6.4

SECCIÓN 7. Manipulación y almacenamiento

- 7.1 Precauciones para una manipulación segura
- Evitar la inhalación de vapor o neblina. Conservar alejado de toda llama o fuente de chispas No
- impedir la acumulación de descargas elctrostáticas. Ver precauciones en la sección 2.2
- Almacenar en un lugar fresco. Conservar el envase hermét seco y bien ventilado. Los contenedores que se abren debe cuidadosamente y mantener en posición vertical para evita Condiciones de almacenamiento seguro, incluidas po 7.2

Manipular y almacenar en atmósfera inerte.

Usos específicos finales 7.3

Aparte de los usos mencionados en la sección 1.2 no se es

SECCIÓN 8. Controles de exposición/protección individua

8.1 Parámetros de control

Componentes con valores límite ambientales de exp No contiene sustancias con valores límites de exposición p

Controles de la exposición

Controles técnicos apropiados 8.2

Manipular con las precauciones de higiene industrial adecu de seguridad. Lávense las manos antes de los descansos y jornada laboral.

Protección personal

Protección de los ojos/ la cara Gafas de seguridad con protecciones laterales confon de protección para los ojos probado y aprobado segú correspondientes, tales como NICSH (EE.UU.) o EN

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		oducto, que presente niveles nógeno humano probable, posible al de Investigaciones sobre			osición única	osiciones repetidas			/estigado adecuadamente las								evaluación de la seguridad					ador apto para productos	sobrante y las soluciones no-		Pagina 6 de 8	Merck
Mutagenicidad en celuias germinales Sin datos disponibles	loge	IARC: No se identifica ningún componente de este producto, que presente niveles mayores que o jugal a lo 1,9% como agente carcinógeno humano probable, posible o confirmado por la (IARC) Agencia Internacional de Investigaciones sobre	Carcinógenos.	Toxicidad para la reproduccion Sin datos disponibles	Toxicidad específica en determinados órganos - exposición única Sin datos disponibles	Toxicidad específica en determinados órganos - exposiciones repetidas	ani dados disponinces Peligo de aspiración Sin datos disponibles	Información Adicional RTECS: 1115025000	Seqún nuestras informaciones, creemos que no se han investigado adecuadamente las	propiedades químicas, físicas y toxicológicas.	SECCIÓN 12. Información ecológica	to t Touloidad	Sin datos disponibles	12.2 Persistencia y degradabilidad Sin datos disponibles	12.3 Potencial de bioacumulación Sin datos disponibles	12.4 Movilidad en el suelo Sin datos disponibles	12.5 Resultados de la valoración PBT y mPmB La valoración de PBT / mPmB no está disponible ya que la evaluación de la seguridad	química no es necesaria / no se ha realizado	12.6 Otros efectos adversos Sin datos disponibles	SECCIÓN 13. Consideraciones relativas a la eliminación	13.1 Métodos para el tratamiento de residuos	Producto Este producto combustible puede quemarse en un incinerador apto para productos	químicos provisto de postquemador y lavador. Ofertar el sobrante y las soluciones no- aprovechables a una compañia de vertidos acreditada.	Envases contaminados Ellminar como producto no usado.	Aldrich- A86406	The life science business of Merck operates as MilliporeSigma in
										dadas.					Productos de descomposición peligrosos Productos de descomposición peligrosos formados en condiciones de incendio Óxidos de	disponibles				obco organização negro. Dobervaciones: Organos de los Sentidos (Olfato, Vista, Oldo y Gusto):Vista: Prosis. Conducta: Sommolendia (depresión egneral de la actividad) Pulmones, torax o Respiración:					Pagina 5 de 8	Merck
										mend					en cond	datos				ta, Oíc ividad						igma in
Sin datos disponibles	Cia datas disessibles	Sin datos disponibles	SIN datos disponibles	Sin datos disponibles	Sin datos disponibles	Sin datos disponibles	Otra información de seguridad Sin datos disponibles	SECCIÓN 10. Estabilidad y reactividad		Estabilidad química Estable bajo las condiciones de almacenamiento recomendadas.	Posibilidad de reacciones peligrosas Sin datos disponibles	Condiciones que deben evitarse	Calor, llamas y chispas.	10.5 Materiales incompatibles Agentes oxidantes fuertes	Productos de descomposición peligrosos Productos de descomposición peligrosos formados e	carbono productos de descomposición peligrosos - Sin datos disponibles En caso de incendio: véase sección 5		SECCIÓN 11. Información toxicológica	11.1 Información sobre los efectos toxicológicos Toxicidad aguda	occo oral rada 2000 myra Doservaciones: Organos de Sentidos (Olfato, Vista, Oído y Gusto):Vista: Ptosis. Conducta: Sommolencia (depresión general de la actividad) Pulmones, torax o Resp		Corrosión o irritación cutáneas Sin datos disponibles	Lesiones o irritación ocular graves Sin datos disponibles	Sensibilización respiratoria o cutánea Sin datos disponibles		The life science business of Merck operates as MilliporeSigma in

	entropy of the second				andiración an admana Ilimiteda	nounceron en numero minicado no pretende ser exhaustiva y	ación contenida en este conocimiento y es aplicable a	o. No representa minguna Sigma-Aldrich y sus suitante de la manipulación o sioma-aldrich.com v/o a los	a o de la nota de entrega. lina de este documento puede as hacemos la transición de umento relativa al producto	ido. Para mas informacion,										Pagina 8 de 8	Merck
	15.2 Evaluación de la seguridad química Dara este producto no se ha llevado a cabo una evaluación de la securidad química	רמום בסוב להסתתרוס זוס סב זום וובאמתה מ רמסה מוום באמוחמרוה		SECCIÓN 16. Otra información	Otros datos Convelsity 2010 Clama Aldelat Co. 110. Co. autocian la sociedada da advinces Illectrado	copyright cuts organization cut cut, se autoriza la reproduction en inimero miniado de copias para uso exclusivamente interno. La información indicada armba se considera correcta pero no pretende ser exhaustiva y	deberá utilizarse únicamente como orientación. La información contenida en este documento esta basade nel presente estado de nuestro conóminiento y es aplicable a la concensiona de contribuída conocidan sera el producto de nuestro de conóminientes en	tes precaucutes de segunda apropatos para es producto. Nu representa mujura grantia de las propredades del producto. La Corporación Sigma-Aldrich y sus Compañías Alindas, no responderán pro ringún daño esultante de la manjulación o contactor no al inordurto indicado artiha. Dinises a xww esultante-aldrich, com Vo a los	términos y condiciones de verta en la reverso de la actua o de la noda de entrega. La marca que apacte en el mostazota y/or el pel apágnia de actua de condición entregues no conoción visualmente con el producto adquinido mientras hacenos la transción de nuestra marca. Sin embargo, toda la información del documento relativa a producto	permanece sin cambios y coincide con el producto solicitado. Para mas informacion, póngase en contacto con misbranding@sial.com										Aldrich- A86406	The life science business of Merck operates as MilliporeSigma in the US and Canada
		IATA: -	es Unidas			ІАТА: -	IATA: -	marino: IATA: no			juridad, salud y medio ambiente	os de la Reglamento (CE) No.	: No prohibido y/o restringido	: No prohibido y/o restringido	: No prohibido y/o restringido	: Este producto no contiene sustancias muy preocupantes (Reglamento (CE) No. 1907/2006 (REAGH), articulo 57).	snexo XIV de la regu lación REACH (CE)	stancia requiere aut o por ejemplo e n analíticas ruti narias o el uso como	: No prohibido y/o restringido	Pagina 7 de 8	Merck
SECCIÓN 14. Información relativa al transporte	14.1 Número ONU	ADR/RID: - IMDG: -	14.2 Designación oficial de transporte de las Naciones Unidas	ADR/RID: Mercancia no peligrosa IMDG: Not dangarous goode		14.3 Clase(s) de peligro para el transporte ADR/RID: - IMDG: -	14.4 Grupo de embalaje ADR/RID: - IMDG: -	14.5 Peligros para el medio ambiente ADR/RID: no IMDS Contaminante marino: IATA: no no	14.6 Precauciones particulares para los usuarios Sin datos disponibles	SECCIÓN 15. Información reglamentaria	15.1 Reglamentación y legislación en materia de seguridad, salud y medio ambiente	especificas para la sustaincia o la mezcia La hoja técnica de seguridad cumple con los requisitos de la Reglamento (CE) No. 1907/2006.	Internacional sobre las Armas CWC) Programas sobre los Productos Xitos y los Precursores (Louisiana ive Code, Title 33, Part V Section eq.)	REACH - Restricciones a la fabricación, comercialización y uso de determinadas sustancias, preparados y artículos peligrosos (Anexo XVII)	Reglamento (CE) n o 649/2012 del Parlamento Europeo y del Consejo relativo a la exportación e importación de productos químicos peligrosos	REACH - Lista de sustancias candidatas que suscitan especial preocupación para su Autorización (artículo 59).	Ese producto contriene una sustancia incluida en el anexo XIV de la regu lación REACH (CE) 0. 1907/2006. Sustanon unuda / Fecha de	Después de la fecha de expiración, el uso de esta sustancia requiere aut o por ejemplo e n la investigación y desarrollo científicos que incluyan análiticas ruti nanas o el uso como intermedio.	Reglamento (CE) no 1005/2009 sobre las	Aldrich- A86406	The life science business of Merck operates as MilliporeSigma in the US and Canada

sustancias que agotan la capa de ozono



FICHA DE DATOS DE SEGURIDAD de acuerdo el Reglamento (CE) No. 1907/2006

SECCIÓN 1. Identificación de la sustancia o la mezcla y de la sociedad o la empresa

1.1 Identificadores del producto

- : v-Valerolactona Nombre del producto
- V403 Marca REACH No. Referencia
- ya que la sustancía o sus usos están exentos del registro, el tonelaje anual no requiere registro o dicho registro está previsto Aldrich Un número de registro no está disponible para esta sustancia, para una fecha posterior 108-29-2
 - Usos pertinentes identificados de la sustancia o de la mezcla y usos No. CAS

desaconsejados 1.2

: Reactivos para laboratorio, Fabricación de sustancias Usos identificados

Datos del proveedor de la ficha de datos de seguridad 1.3

Merck Life Science S.L. Calle Maria de Molina 40 E-28006 MADRID	+34 916 619 977	+34 916 619 642	serviciotecnico@merckgroup.com
Compañía	Teléfono	Fax	E-mail de contacto

Teléfono de emergencia 1.4

Teléfono de Urgencia	 E CH
	+(34)-931768545 (CHEMTREC
	internacional)

(CHEMTREC		
+(34)-931768545	internacional)	

SECCIÓN 2. Identificación de los peligros

2.1 Clasificación de la sustancia o de la mezcla

- No es una sustancia o mezcla peligrosa.
- Elementos de la etiqueta

2.2

No es una sustancia o mezcla peligrosa.

Otros Peligros 2.3

Esta sustancia/mezcla no contiene componentes que se consideren que sean bioacumulativos y toxicos persistentes (PBT) o muy bioacumulativos y muy persistentes (PME) a invises del 0,1%, o superiores.

Aldrich- V403

The life science business of Merck operates as MilliporeSigma in the US and Canada



SECCIÓN 3. Composición/información sobre los componentes Sinóni 3.1

y-VALEROLACTONE

ancias	
nimos	: (±)-γ-Valerolactone
	y-Methyl-y-butyrolactone
	4,5-Dihydro-5-methyl-2(3H)-furanor
	4-Hydroxypentanoic acid lactone
	gamma-Valerolactone

e

Formula	: C5H8O2	
Peso molecular	: 100,12 g/mol	
No. CAS	: 108-29-2	

: 203-569-5 Peso mole No. CAS No. CE

Según la normativa aplicable no es necesario divulgar ninguno de los componentes.

SECCIÓN 4. Primeros auxilios

4.1 Descripción de los primeros auxilios

Consultar a un médico. Mostrar esta ficha de seguridad al doctor que esté de servicio. Recomendaciones generales

Si es inhalado

Si aspiró, mueva la persona al aire fresco. Si ha parado de respirar, hacer la respiración artificial. Consultar a un médico.

En caso de contacto con la piel Eliminar lavando con jabón y mucha agua. Consultar a un médico.

En caso de contacto con los ojos

Lavarse abundantemente los olos con aqua como medida de precaución. Por ingestión

Nunca debe administrarse nada por la boca a una persona inconsciente. Enjuague la boca

Principales síntomas y efectos, agudos y retardados Los síntomas y efectos más importantes conocidos se describen en la etiqueta (ver sección con agua. Consultar a un médico. 4.2

- Indicación de toda atención médica y de los tratamientos especiales que deban 2.2) y / o en la sección 11 4.3
 - dispensarse inmediatamente Sin datos disponibles

SECCIÓN 5. Medidas de lucha contra incendios

5.1 Medios de extinción

Usar agua pulverizada, espuma resistente al alcohol, polvo seco o dióxido de carbono. Medios de extinción apropiados

Peligros específicos derivados de la sustancia o la mezcla 5.2

Oxidos de carbono

- Recomendaciones para el personal de lucha contra incendios Si es necesario, usar equipo de respiración autónomo para la lucha contra el fuego. 5.3

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	del Reglamento (UE) 2016/425 y de la norma EN 374 derivada del mismo.
SECCIÓN L LING CO P P P P P P P P P P P P P P P P P P	
1 1 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2	nm L 740 / Aldrich Z677272, Talla M)
SECCIÓN HIT PODO SECCIÓN SECCI	nm L 740 / Aldrich Z677272, Taila M)
9 SECCIÓN 1 INÓ 9 C C C S 1 T C C C S 1 T C C C S 1 T C C C C C S 1 T C C C C C S 1 T C C C C C C C S 1 T C C C C C C C S 1 T C C C C C C C C C C C C C C C C C C	51.24 Eichenzell, Teléfono +49 (0)6659 87300, e- ebbs Can Oras sustancias, y hajo condiciones en contras sustancias, y hajo condiciones en contrastico con el prosector de los guartes los martes conseptible y Merce Sare suprato e hajene inclusio. No deer autorito suprato entre contentes, en vo deer autorito con la supratoritor con el prosector de los pro- supratoritor con el prosector de los pro- supratoritor con el prosector de los pro- toritoritor de los pro- toritoritoritoritoritoritoritoritoritori
9 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5	to de equipamiento de protección debe ser elegido tad de sustancia pelígrosa al lugar específico de
9 1 1 1100 9 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2	n muestre que los respiradores purificadore toda la solto (EEUU) o tipo AEEK (EN 14387/ ingienena. Si usar un respiradores y suar respiradores y sios bajo los estandards gubernamentales
SECCIÓN 9. Propledades físic: 9.1. Información sobre propi a) Aspecto b) Olor b) Olor c) Umbral offativo d) pH Punto de ongadadin f) Punto intervalo de ebilidión f) Punto intervalo de ebilidión f) Tasa de evaporación h) Tasa de evaporación	a CEN (UE) iai N el sistema de alcantarillado.
 3.1 Información sobre propi a) Aspecto b) Olor b) Unbral offativo d) pH Punto de fusión/ punto intervalo de bullidon intervalo g) Punto de infamación h) Tasa de evoproración 	as
 Aspecto Olor Umbral olfativo Umbral olfativo Punto de fusión/ Punto ale fusión/ Punto intervalo Punto de inflamación Punto de inflamación 	icas y químicas básicas o transfe
 b) Olor c) Umbral olfativo d) pH Punto de fusión/ punto de naglación punto intervalo de eximición entervalo g) Punto de inflamación 	o, inquitato oro
 c) Umbrai olfativo d) pH e) Punto de fusión/ punto de congradión f) Punto inicial de de eluilición e infermación g) Punto de infermación 	sponibles
 e) Punto de fusión/ punto de orgélación f) Punto inicial de e e e ebulición e intervalo de ebulición g) Punto de inflamación h) Tasa de evaporación 	sponibles sponibles
 Punto inicial de e bullicón e transaio de bullicón g) Punto de inflamación h) Tasa de evaporación 	valo de fusión: -31 °C - lit.
g) Punto de inflamaciónh) Tasa de evaporación	a 10 mmHg - lit. C - lit.
II) I ASA UE EVADORACION	a cerrada
	sponibles
Pagina 3 de 8 Aldrich- V403	Pagina 4 de 8
The life science business of Merck operates as MilliporeSigma in the US and Canada	liporeSigma in

Sin datos disponibles **Otros datos** 5.4

SECCIÓN 6. Medidas en caso de vertido accidental

- 6.1 Precauciones personales, equipo de protección y proce Utilicese equipo de protección individual. Evitar respirar los v Asegúrese una ventilación apropiada. Equipo de protección individual, ver sección 8.
- Precauciones relativas al medio ambiente No dejar que el producto entre en el sistema de alcantarillado 6.2
 - Métodos y material de contención y de limpieza 6.3
- Empapar con material absorbente inerte y eliminar como un contenedores apropiados y cerrados para su eliminación.
 - Referencia a otras secciones Para eliminación de desechos ver sección 13. 6.4

SECCIÓN 7. Manipulación y almacenamiento

- 7.1 Precauciones para una manipulación segura Ver precauciones en la sección 2.2
- Condiciones de almacenamiento seguro, incluidas posil Almacenar en un lugar fresco. Conservar el envase hermética 7.2
- seco y bien ventilado.
 - 7.3
 - Usos específicos finales Aparte de los usos mencionados en la sección 1.2 no se estip

SECCIÓN 8. Controles de exposición/protección individual

8.1 Parámetros de control

Componentes con valores límite ambientales de exposi No contiene sustancias con valores límites de exposición prof

Controles de la exposición 8.2

Controles técnicos apropiados

Manipular con las precauciones de higiene industrial adecuad de seguridad. Lávense las manos antes de los descansos y de jornada laboral.

Protección personal

Protección de los ojos/ la cara

Gafas de seguridad con protecciones laterales conforme de protección para los ojos probado y aprobado según la correspondientes, tales como NIOSH (EE.UU.) o EN 166

Protección de la piel

Manipular con guantes, Los guantes deben aer inspeccio Utilice la técnica correcta de quitarse los guantes (sin to guante) para evitar el contacto de la piel con este produ contaminados después de su usos, de conformidad con la prácticas de laboratorio. Lavari y secar las manos.

Aldrich- V403

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	in the second	colligad muscular Pulmones,							ucto, que presente niveles geno humano probable, posible de Investinaciones cobre			ición única	iciones repetidas				stigado adecuadamente las							Pagina 6 de 8	Merck
SECCIÓN 11. Información toxicológica	 Intermación sobre los efectos toxicológicos Toxicidade aguda Dució Orar - Rata - 8,800 mg/kg 	Observatories: Conjoucia: Arrestesto general. Conjoucia: Jue torax o Respiratoria: Estimulación respiratoria DL50 Cutáneo - Coneio - > 5.000 mo/kg	Corrosión o irritación cutáneas	Piel - Conejo Resultado: Ligera irritación de la piel - 24 h	Lesiones o irritación ocular graves Sin datos disponibles	Sensibilitzación respiratoria o cutánea Sin datos disponibles	Mutagenicidad en células germinales Sin datos disponibles	ő	IARC: No se identifica ningún componente de este producto, que presente niveles mayores que o jogual a 0,1% como agente carrindgeno humano probable, posible o confirmado nor la (IABC). Anancia intermedinal de Investinaciones cotras	Carcinógenos.	Toxicidad para la reproducción Sin datos disponibles	Toxicidad específica en determinados órganos - exposición única	Toxicidad específica en determinados órganos - exposiciones repetidas	Sin datos disponibles	Peligro de aspiración Sin datos disponibles	Información Adicional RTECS: LU3580000	Según nuestras informaciones, creemos que no se han investigado adecuadamente las	propreudues quimicas, insicas y toxicologicas.	SECCIÓN 12. Información ecológica	12.1 Toxicidad Sin datos disponibles	12.2 Persistencia y degradabilidad Sin datos disponibles	12.3 Potencial de bioacumulación Sin datos disponibles	12.4 Movilidad en el suelo Sin datos disponibles	Aldrich- V403	The life science business of Merck operates as MilliporeSigma in the US and Canada
																dadas.				Productos de descomposición peligrosos Productos de descomposición nelicrosos formados en condiciones de incendio - Óvidos de	s disponibles			Pagina 5 de 8	Merck
Sin datos disponibles	Sin datos disponibles	Sin datos disponibles	1,05 gcm3 a 25 °C	Sin datos disponibles	log Pow: -0,133	Sin datos disponibles	Sin datos disponibles	Sin datos disponibles	Sin datos disponibles	Sin datos disponibles	guridad	3,46 - (Aire = 1.0)		actividad		Estabilidad química Estable bajo las condiciones de almacenamiento recomendadas.	es peligrosas	evitarse	S	ssición peligrosos ión peligrosos formados en cor	ortros productos de descomposición peligrosos - Sin datos disponibles	e sección 5			perates as MilliporeSigma in
i) Inflamabilidad	 j) Inflamabilidad superior/inferior o limites explosivos 	 k) Presión de vapor l) Densidad de vapor 	_	n) Solubilidad en agua	 o) Coeficiente de reparto n- octanol/agua 	 p) Temperatura de auto-inflamación 	 q) Temperatura de descomposición 	r) Viscosidad	s) Propiedades explosivas	t) Propiedades	9.2 Otra información de seguridad	Densidad relativa del 3,46 - (Aire = 1.0) vanor		SECCIÓN 10. Estabilidad y reactividad	10.1 Reactividad Sin datos disponibles	10.2 Estabilidad química Estable bajo las condicione	10.3 Posibilidad de reacciones peligrosas Sin datos disponibles	10.4 Condiciones que deben evitarse Sin datos disponibles	10.5 Materiales incompatibles Agentes oxidantes fuertes	10.6 Productos de descomposición peligrosos Productos de descomposición peligrosos form	carbono Otros productos de descor	En caso de incendio: véase sección 5		Aldrich- V403	The life science business of Merck operates as MilliporeSigma in the US and Canada

Esta sustancia/mezcla no contiene componentes que se consideren que sean blocumulativos y foxicos persistentes (PBT) o muy bloacumulativos y muy persistentes (PPD) a mixeles del 0,1% o superiores.

12.6 Otros efectos adversos

Sin datos disponibles

SECCIÓN 13. Consideraciones relativas a la eliminación

13.1 Métodos para el tratamiento de residuos

Producto Ofertar el sobrante y las soluciones no-aprovechables a una compañia de vertidos acreditada.

Envases contaminados Eliminar como producto no usado.

SECCIÓN 14. Información relativa al transporte

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	•		

IATA: -IMDG: ADR/RID: -

- Designación oficial de transporte de las Naciones Unidas ADR/RID: Mercancía no peligrosa 14.2
 - Not dangerous goods Not dangerous goods IATA:

IATA: -14.3 Clase(s) de peligro para el transporte IMDG: 14.4 Grupo de embalaje ADR/RID:

IMDG: -ADR/RID:

IATA: -IATA: no

- 14.5 Peligros para el medio ambiente ADR/RID: no IMDG Contaminante marino:
 - 00

14.6 Precauciones particulares para los usuarios

Sin datos disponibles

SECCIÓN 15. Información reglamentaria

15.1. Reglamentación y legislación en materia de seguridad, salud y medio ambiente especificas para la sustancia o la mezcla La floja fectiva de seguridad cumple con los requisitos de la Reglamento (CE) No.

1907/2006.

15.2

Evaluación de la seguridad química Para este producto no se ha llevado a cabo una evaluación de la seguridad química

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SECCIÓN 16. Otra información

Otros datos

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deberá utilizarse únicamente como orientación. La información contenida en este documento esta basada en el presente estado de nuestro conocimiento y es aplicable a La marca que aparece en el encabezado y/o el pie da página de este documento puede no conciertir vasamente con el protocto acturinto menteras hacenación de nuestra marca. Sin embago, toda la información del documento relativa a producción entremenes na cuandos y concider en el producto solicidado. Para más información, pórgase en contacto con mitióngiesal.com garantia de las propiedades del producto. La Corporación Sigma-Aldrich y sus Compañías Aliladas, no responderán por ningún dalor exultante de la manjudiación o contacto con el producto indicado arriba. Dirijase a www.sigma-aldrich.com y/o a los términos y condiciones de vente en el reverso de la factura o de la nida de entrega. las precauciones de seguridad apropiadas para el producto. No representa ninguna

Pagina 8 de 8 **Zerck**

2/6/2020	HYDROGEN

CAS #: 1333-74-0 UN #: 1049 EC Number: 215-605-7

ICSC 0001 - HYDROGEN

	ACUTE HAZARDS	PREVENTION	FIRE FIGHTING
FIRE & EXPLOSION	FIRE & Externely filammable, Marry reactors may cuese fire or explosion. Castair mixtures are explosion.	Shurd fair specify fair opcashe and No open filmmes, NO sparks and NO for rak to surroundings, lidit this strongly, Doads system, or rank to surroundings. In other care, power the strong strong strong strong strong strong strong power exploring and signifying. No encom- ention and signifying the encom- strong	Shut off supply, if not possible and ot on stark to surrounding, let the fire burn itself out, in other cases all exitinguish with water strary, powder, carbon dioxide. In case of fire, keep cylinder cool by straying with water Combat fire from a shellered position.

	Use appr	Use appropriate engineering controls.	
	SYMPTOMS	PREVENTION	FIRST AID
Inhalation	Dizziness, Headache, Lethargy, Suffocation.	Use ventilation.	Fresh air, rest.
Skin	ON CONTACT WITH GAS: FROSTBITE.	Cold-insulating gloves.	ON FROSTBITE: rinse with plenty of water, do NOT remove clothes. Refer immediately for medical attention.
Eyes	ON CONTACT WITH GAS: FROSTBITE.	Wear face shield.	ON FROSTBITE: rinse with plenty of water. Refer immediately for medical attention.
Ingestion			

CLASSIFICATION & LABELLING	According to UN GHS Criteria		Extremely flammable gas	contains gas under pressure; may explode in reated transportation	un classification UN Hazard Class: 2.1	on behalf of ILO and WHO.
SPILLAGE DISPOSAL	Evacuate danger area! Consult an expert! Ventilation. Remove all ignition sources. Remove vapour with fine water spray.	STORAGE	Fireproof. Cool. Ventilation along the floor and ceiling. Separated from oxidizing materials.	PACKAGING		Model M

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ICSC: 0001			
	ICSC: 0001		

HYDROGEN

ICSC 0001 - HYDROGEN

22/6/2020

ICSC: 0001 April 2014

UVDDOCEN	1000 .2321
HTURUGEN	1020: 0001
PHYSICAL & CHEM	PHYSICAL & CHEMICAL INFORMATION
Physical State: Apparance (000URLISS COLOURLESS COUPRESSED GAS. Physical angers Physical angers Physical angers Physical angers Physical and angers Physical and Physical Angels Physical Angels Physical Angels Physical Angels Physical Angels Physical Physical Angels Physical Physical Physical Physical Physical Physical Physical Physical Physical Physical Physical Physical Physical Physical Physical Physica	Formula: H2 Belling point: -555:C Belling point: -555:C Relative voyacut offensity (int = 1): 0.07 Relative voyacut offensity (int = 1): 0.07 Auto-ballion (interventing 55):C
Chemical angles's Behavioral angles's Behavioral and angles's Behavioral and angles's and grade and and and particular sounds you and and peak preference these reactions, peak preference these reactions.	Explosive limits, volfs in air. 4-75 Vapour presevue, kª ba ai 25°C: 16520 Solublity in water, mg/ ai 21°C: 122 (very poor)
EXPOSURE & H	EXPOSURE & HEALTH EFFECTS
Routes of expensio Exposure meinly occurs via inhalation. Expected of short-term exposure Reflected of short-term exposure Archynation. See Notes. Exposure to cold gas could cause freatible.	Inhibition risk. Inhibition risk. Deverge the oxygen coveral of the air in confried areas. Effects of long-term or repeated exposure
OCCUPATIONAL I	OCCUPATIONAL EXPOSURE LIMITS
ENVIRG	ENVIRONMENT
ON	NOTES
Heb-constructions in the strates deficiency of oxygen with the risk of uncorrectourness or death. News cognition constructions with suitable gas detector (a normal farmmable gas detector is NOT suitable for the purpose).	he risk of unconsciousness or death. ormal flammable gas detector is NOT suitable for the purpose).
ADDITIONAL	ADDITIONAL INFORMATION
EC Classification Symbol: F+; R: 12; S: (2)-9-16-33	

1/2

2/2



Uso recomendado Usos desaconsejados

Empresa

Nombre del producto

Fórmula molecular

nónimos . CAS . CE.

exto completo de las indicaciones de peligro: ver la sección 16

SECCIÓN 4: PRIMEROS AUXILIOS

ACR12514

A la vista de los datos disponibles, no se cumplen los criterios de clasificación

Peligros físicos

Página 1/10

ACR12614

LEVULINIC ACID

Página 2/10

Levulinic acid	Ficha DE DATOS DE SEGURIDAD Fecha de revisión 2046-2019	FICHA DE DATOS DE SEGURIDAD Fecha de revisión 2046-2019
4.1. Descripción de los primeros auxilios	auxilios	No debe liberarse en el medio ambiente.
Consejo general	Si persisten los sintomas, liamar a un médico.	6.3. Metodos y material de contención y de limpleza
Contacto con los ojos	Enjuagar inmediatamente con abundante agua, también bajo los párpados, durante al menos 15 minutos. Consultar a un médico.	Mantener en contenedores carrados aptos para su eliminación. Recoger o aspirar el derrame y ponerío en un contenedor adecuado para la eliminación.
Contacto con la piel	Lavar immediatamente con abundante agua durante al menos 15 minutos. Si persiste la intración cutánea, llamar a un médico.	6.4. Referencia a otras secciones.
Ingestión	Limpiar la boca con agua y beber a continuación abundante agua. Consultar a un médico si se producen sintomas.	Consultar las medidas de protección en las listas de las secciones 8 y 13.
Inhalación	Sacar al aire libre. Si no respira, realizar técnicas de respiración artificial. Consultar a un médico si se producen sínhomas.	SECCIÓN 7: MANIPULACIÓN Y ALMACENAMIENTO
Equipo de protección para el personal de primeros auxilios	Asegurares de que el personal médico sea consciente de los materiales implicados, tomado procauciónes para protegense a sí mismos y para evitar extender la	7.1. Descuelents para una manipulación segura. Levar explose de pretección inductal: Envira el contacto con los ejos, la pel o la ropa. Asegurar una vertilación adecuada. Evitar descripción y la prepetición factoria formación de polo:
4.2. Principales sintomas y efectos, agudos y retardados	os, agudos y retardados	ta a ti ta fina da ser
	Ninguno razonablemente predecible.	menues myennes Manipular respeatando las buenas prácticas de higiene industrial y seguridad. Manténgase lejos de alimentos, bebúdas y piensos. No comer, beber ni fumar durante su utilización. Quitar y lavar la ropa contarminada antes de reutilizar. Lávense las manos antes
4.3. Indicación de toda atención r	4.3. Indicación de toda atención médica y de los tratamientos especiales que deban dispensarse inmediatamente	de los descansos y después de terminar la jornada laboral.
Notas para el médico	Tratar los síntomas.	7.2. Condiciones de almacenamiento seguro, incluidas posibles incompatibilidades
SECCI	SECCIÓN 5: MEDIDAS DE LUCHA CONTRA INCENDIOS	Mantener en un lugar fresco, seco y bien ventilado. Mantener el recipiente herméticamente cerrado. Mantener alejado de la luz directa del sol.
6.4 Madian da autimaión		7.3. Usos específicos finales
		Uso en laboratorios
Medios de extinción apropiados Usar agua pulverizada, espuma res	Medios de extinción apropiados. Usar agua puiverizada, espuma resistenta al alcohol, polvo seco o dióxido de carbono.	SECCIÓN 8: CONTROLES DE EXPOSICIÓN/PROTECCIÓN INDIVIDUAL
Medios de extinción que no debe No hay información disponible.	Medice de extinción que no deben utilizarse por razones de seguridad No hay información disponible.	8.1 Parámetros de control
5.2. Peligros específicos derivados de la sustancia o de la mezcla	os de la sustancia o de la mezcla	Limites de exposición
Su descomposición térmica puede -	Su descomposición térmica puede dar lugar a la liberación de vapores y gases irritantes.	Este producto, tal y como se ha suministrado, no contiene ningun material peligroso con limites de exposicion laboral establecidos por las organismos reguladores específicos de la región
Productos de combustión peligrosos Monóxido de carbono (CO), Dióxido de carbono (CO2)	asas o de carbono (COZ).	
5.3. Recomendaciones para el pe	5.3. Recomendaciones para el personal de lucha contra incendios	
Como en cualquier incendio, llevar equivalente) y todo el equipo de pro	Como en cualquier incendio, llevar un aparato de respiración autónomo de presión a demanda MSHANNOSH (aprobado o equivalente) y todo el equipo de protección necesario.	
SECCIÓN	SECCIÓN 6: MEDIDAS EN CASO DE VERTIDO ACCIDENTAL	Vatores timite biologores suministra, no contiene ringún material peligroso con límites biológicos establecidos por los organismos reguladores regionales especificos
6.1. Precauciones personales, eq	6.1. Precauciones personales, equipo de protección y procedimientos de emergencia	
Asegurar una ventilación adecuada	Asegurar una ventilación adecuada. Utilicese equipo de protección individual. Evitar la formación de polvo.	Métodos de seguimiento
6.2. Precauciones relativas al medio ambiente	die ambiente	EN 14042 2003 Titulo de identificación: Atmósferas en los lugares de trabajo. Directrices para la aplicación y uso de
ACR12514	Pagina 3/10	ACR12514 Pagina 4/10

Levulinic Acid upgrade to y-valerolactone by hydrogenation by using a bifunctional catalyst.

Nivel an effecto derivado (DNEL) No hay información disponible <u>Reta de supeisición</u> Effecto agudo (local) Effecto agudo (local) (esterimica) Carteres Indeatodin Contentración prevista sin efecto. No hay información disponible. PREC)	SEC	
témica)		ercción o, propinante rísicas y ouitaicas
Oral Cutana Intralición Amententedion previeta sin efecto No hay información disponible. MEC		ION 9: FROFIEDADES FISICAS 1 QUIMICAS
Inhalación neemtración prevista sin efecto No hay información disponible. NEC)	9.1. Información sobre propiedades físicas y químicas básicas	fisicas y químicas básicas
ncentración prevista sin efecto No hay información disponible. VEC)	Aspecto Estado físico	Amarillo Sólido con bajo punto de fusión Sólido
	Olor Umbrai offativo Puntviintunalo dia fusika	Inadoro No hay datos disponibles 13 - 54 - 64 - 64 - 64 - 64
8.2 Controles de la exposición	Punto de reblandecimiento Punto linterato de abuilición	
Modula Networks Negratur Modulación Antoniada, especialmente en livera continuidas. Alegurares de que haya estacones de laveido de opoi y Costate de segurados de la uchacidad esteciante esta de contro la les contro el alatamiento o confinamiento de proseso. Semeros de las positivos, dostarán entradas se contro la les contro a la subminiento o confinamiento de proseso.		88 °C / 2018 4°F Methods - No hay información disponible No say intomación disponible No say información disponible No hay información disponible
la introducción de cambios en el proceso o los equipos para reducir al mínimo la liberación o el contacto, y el uso de sistemas de ventilación adecuadamente diseñados, dirigidas a controlar los materiales pelígrosos en su fuente.		2 °C
Equipos de protección personal Protección de los ojos Antiparras (Norma de la UE - EN 195)	Densidad relativa / Densidad Densidad relativa / Densidad Densidad aparente	No by datos disponibles
Protección de las manos Guantes protectores	Solucinade en el agua sol Solucidad en ortos dísolventes No Coeficiente de resario (n-octanol/acua)	souche No hay información disponible nual
Esi		No hay datos disponibles
Caucho ratural peretración guartes EN 374 (requisito mínimo) Caucho ratural roccinadas e consulta de monte entración de laboradares como a de laboradares Necesarios	10) International Internation Viscosidad everyologivas Propledades comburentes	No es aplicable Solido No as aplicable Solido No hay información disponible No hay información disponible
PVC Protección de la piel v el cuerto Ropa de manoa larca	9.2. Otros datos	
impeccione los guartes artes de su uso. Testamio Lorens de la factoriante na una de la permedidada y el tempo de adelanto que son proveitos por el proveedor de Asguartes dor son os guartes atom adecados para la termación.	Fórmula molecular Peso molecular	C5 H8 03 116 12
química compatibilidad, destreza, condiciones de funcionamiento en También tiene en cuenta las condiciones locales espectificas bajo las cuales el producto es utilizado, tal como el diuses los quantes con rudados para evidar contaminación de la piel.	SE	SECCIÓN 10: ESTABILIDAD Y REACTIVIDAD
Protección respiratoria Cuando los trabajadores se enfrentan a concentraciones superiores al límite de exposición deben utilizar restinadores centificados aconcisados.	t de exposición,	Ninguno conocido, en base a la información facilitada
Para protegior a quien to livera, el equipo de protección respiratoria debe ajustarse conrectamente y estar sometido a un uso y un mantenimiento adecuados	slarse 10.2. Establildad química	Estable en condiciones normales.
A gran escala / uso de emergencia Utilico un NIOSH / MSHA o la norma europea EN 136 respirador aprobado si los límites de experiente de exposición en excaedidos rimitación con entración o traisción totas sintomas sont experimentados		grosas
Inpolar interview interview interview interview interview interview interview and interview and interview intervi	Polimerización peligrosa Reacciones peligrosas	No se produce ninguna polimerizacion peligrosa. Ninguno durante un proceso normal.
limites de exposición son excedidos o irritación u otros síntiomas son experimentados Recomendado máscana: - Válvula de fitrado: EN405, o, Media máscana: EN405, con fitro, ES141, endo	nentados scara: EN140; evitarse	Exposición a la luz. Productos incompatibles.
AI EPR se utiliza una prueba de ajuste de la máscara debe lievarse a cabo	10.5. Materiales incompatibles	Bases, Agentes oxidantes fuertes, Agentes reductores.

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F Levulinic acid	FICHA DE DATOS DE SEGURIDAD	F Levulinic acid	FICHA DE DATOS DE SEGURIDAD Fecha de revisión 20-46b-2019
10.6. Productos de descomposición	10.6. Productos de descomposición peligrosos. Monóxido de carbonio (50). Dióxido de carbonio (502).	S	SECCIÓN 12: INFORMACIÓN ECOLÓGICA
SEC	SECCIÓN 11: INFORMACIÓN TOXICOLÓGICA	12.1. Toxicidad	No liver for residing ne ner al dasseria.
11.1. Información sobre los efectos toxicológicos	toxicelógicos.		
Información del producto		12.2. Persistencia y degradabilidad Persistencia	Miscible con anua a pereistencia es improbable en base a la información facilitada
(a) toxicidad aguda; Oral Cutánea Inhalación	Categoria 4 14 vistas des datos disponibles, no se cumpion los criterios de clasificación A la vistas de los datos disponibles, no se cumpion los criterios de clasificación	Soluble en agua 12.3. Potencial de bioacumulación. La bioacumulación es improbable	Soluble en agua. La bioacumulación es improbable
Componente Ácido pentanóico, 4-oxo-	DL50 Onal DL50 utitime LC50 inhatoción LD50 mg/kg (Rat) LD50 > 5 g/kg (Rabbit) LC50 inhatoción	12.4. Movilidad en el suelo	E producto es soluble en agua y puede propagarse en sistemas acuosos Probablemente
(b) corrosión o irritación cutáneas;	Categoría 2		sera movil en el medio ambiente dedido a su soubilidad en agual. Autamente movil en suelos
(c) lesiones o irritación ocular graves:	Categoria 2	12.5. Resultados de la valoración PBT y mPmB	No hay datos disponibles para la evaluación.
(d) sensibilización respiratoria o cutánea; Respiratorio No ha Piel No ha	istines: No hay datos disconbies No hay datos disconbies	12.6. Otros efectos adversos Información del alterador del sistema endocrino Contaminantes Orgánicos Persistentes	Este producto no contiene inigún alterador del sistema endocino conocido o sospechoso de serio Este producto no conteiren inigún conocido o suspechado sustancia
(e) mutagenicidad en células germinales;	No hay datos disponibles	Pe	tection de ozono Este producto no contierre mingún conocido o sospechado sustancia SECCIÓN 13: CONSIDERACIONES RELATIVAS A LA ELIMINACIÓN
(f) carcinogenicidad;	No hay datos disponibles Este arrede este en constituente en interiore economidate anno anticipitantes.	13.1. Métodos para el tratamiento de residuos	e residuos
control data la reproducción: No hav datos discorribles	care provocu io unitere uniportentes quimuos recollocudos unito del cargo los No hav datos discontibiles	Desechos de residuos / producto n utilizado	Desectos de residuos / producto notios desectos están clasificados como peligrosos. Dispóngase de acuerdo a las Directivas cutilizado momantivas locales.
(h) toxicidad especifica en	No hay datos disponibles	Embalaje contaminado	Deshâgase de este recipiente en un punto de recogida de residuos especiales o peligrosos.
determinados organos (S IUI) – exposición única;		Catálogo de Desechos Europeos	Según el Catálogo de Desechos Europeos, los Códigos de Desecho no son específico al producto, pero específicos a la aplicación.
 (i) toxicidad especifica en determinados órganos (STOT) – exposición repetida; 	No hay dates disponibles	Otra Información	utilization della sustanti ordigara del acciulos basalandos en la apricación para la que se utiliza el produzio. No tarar ordigara del residuos para el deseguia. No el imitmar el deserto en el a acantanillado. Grandas cantidades afectarán al pH y poducirán darfos en los organismos acutáricos:
Órganos diana	Ninguno conocido.	SECCIÓN	SECCIÓN 14: INFORMACIÓN RELATIVA AL TRANSPORTE
(j) peligro de aspiración;	No es aplicable Skildo	IMDG/IMO	No regulado
Otros efectos adversos	No se han estudiado completamente las propledades toxicológicas.	14.1. Número ONU 14.2. Designación oficial de transporte de las Naciones Unidas	
Sintomas / efectos, agudos y retardados	No hay información disponible	14.3. Clase(s) de peligro para el transporte	
ACR12514	Págha 7/10	ACR12514	Pagina 8/10

Levulinic acid Fecha de revisión 20-feb-2019	Levulinic acid	Fecha de revisión 20-feb-2019
14.4. Croups die embelleje. ADR	Texto completo de las indicaciones H mencionadas en las secciones 2 y 3 1022 - Nocivor en coro en apesión H316 - Provoca irritación cuátera H316 - Provoca irritación cuátras	ecciones 2 y 3
14.1. Número ONU 14.2. Designación oficial de	Leve	Leyenda
transporte de las Maciones Unidas 14.3. Classios de seligro para el transporte		TSCA - Ley de control de sustancias tóxicas (Toxic Substances Control Act) estadounidense, apartado 8(b), inventario DSLNDSL - Lista de sustancias domésticas/no domésticas de Canadá
outpo de emparaje	notificadas notificadas PICCS - Inventario de productos químicos y sustancias químicas de	ENCS - Inventario japonês de sustancias químicas existentes y nuevas
14.1. Nove ONU 14.1. Names ONU 2.1. Desimeration of the	IECSC - inventario chino de sustancias químicas existentes KECL - Sustancias químicas existentes y evaluadas de Corea II	AICS - Inventario australiano de sustancias químicas (Australian Inventory of Chemical Substances) NZIOC - Inventario de productos químicos de Nueva Zelanda
transporte da las Maciones Unidas. 14 activitados de las ferrados en las constantes da las constantes. 14 d. Strong de embaixados en las constantes das constantes d	lesionales f Governmental Industrial Hygienists nistas Industriales Gubernamentales)	TWA - Tiempo Promedio Ponderado IARC - Agencia Internacional para la Investigación del Cáncer
14.6. Freilgros para el medio. No hay peligros identificados imbibierio.	DNEL - Nivel obtanició sin efecto Reperatoria en efecto Reperatoria entreción respiratoria LCB0 - Concentración falal SON NOEC - Concentración sin efecto observado ROEC - Concentración sin efecto observado	PNEC - Concentración prevista sin efecto LDB0 - Dossa: Leals 60% ECS0 - Concentración efectiva 50% POW - Coeficiente de reparto octanol: agua
14.6. Precauciones particulares para lo se requieren precauciones especiales ne recurários	PB1 - Persistemes, bloacumulativas, toxicas	VPVB - Muy persistente y muy pixacumutable
 The instance a granted com. No aplicable, productos envasados entregio a mareos 10 de del Construction. 	ADR: A cuerdo servejo sobre el transport el immicand de mercicia is ADOMAY. Enternicos Carlo Organization/International Au MONDAY: Enternicion Municimo Particulario/International Autime Margineto - Comonio Namescuel para Proveri la Continuación para Dangeresa Carlos Carlos Municimo Paralicitorianistica al Autime Margineto - Comonio Namescuel para Proveri la Continuación para Dangeresa Carlos Carlos Municimo Paralicitoria de Paramito Dangeresa Carlos Carlos Municimo Paralicitoria de Paramito ARE: Entimesion de la programa para para DECE: D'Ametición para la consectión y a Paramito	ADDMATA. Threational Civel Avlation Organization/International Air Transport Association INTRAPOL - Convenio Internacional para Prevenir la Contaminación por los Bruques ATE - Estimacción de la toxicidad a guda
SECCIÓN 15: INFORMACIÓN REGLAMENTARA 15.1. Reglamentación y legislación en materia de seguridad, salud y medio ambiente específicas para la sustancia o la mecióa.	- reactive sourcementanty reproductionation of the component of the com	saaanoo eeyaa dagaadaa oo o
Invention X = enumeran. Companie X = enumeran. Companie ENCE EUCE EUCE EUCE ECC ACG ECC ECC ECC ECC	Conversión de l'encruación Formanyo de l'encruación Formanyo de la provinción preserval a participanta encruación de la preserva de protección preservar a regiment a regimenta de la protección protección compatibilidad, untranse de prenetración, cualados, Usos de preservanta de la protección preserval, a convecta exercica elección, compatibilidad, untranse de prenetración, cualados, preservanta de autoritoria de preservanta de la propoción a productor quintico, includio el uso de restaccives de la veració en de la protección preservanta de autoritoria de la explosición a productor quintico, includio el uso de restaccives de lavera de veración de preservanta de la preservanta de la propoción a productor quintico, includio el uso de restaccives de lavera de veración.	o eliquetado, fichtas de datos de seguridad, equipos de protecc elección, competibilidad, umbrales de penetración, cuidados, cos, incluido el uso de estaciones de lavado de cios y duchas r
Reglamentos nacionales Commonenta Ammunia Clauffezción de las Acuas (VeVVeG) Ammunia - TA.Len Class	Fecha de proparación 17-sep-2014 Fecha de revisión 20-teb-2019 Resumen de la revisión No es aplicable.	
-0x0-	La hoja técnica de seguridad cumple con los requisitos del Reglamento (CE) No. 1907/2006	requisitos del Reglamento (CE) No. 1907/20
15.2. Evaluación de la seguridad química. Un Seguridad Ouímica Evaluación / informe (CSA / CSR) no se ha llevado a cabo	Descargo de responsabilidad La información fucilitada en esta Ficha de Deos de Sponsabilidad su publicación. Dicha información está concedida encorrectar a a muestro leal asber y entender, en la fecha de su publicación. Dicha información está concedida incremente como guía para la seguridad en hamiputución, el tuxo, el processamiento a información esta formación su la función y la la tensor on edistinos formarias con- espectimicación de calidades. La información se reflere unicamient la material supeticionade y puede no ser vidida para tal material testo en combinación se reflere unicamient la material supetico mencionado y pode no ser espectificación de calidades. La información se reflere unicamient la material supetico mencionado y pode no ser espectifique consestententes no conseguiera nora materiales e en cualquier proceso salvo que se	Descripto de responsabilidad Descripto de responsabilidad Entro de la parte la sector lea laber y entender, en la fech calcarrentes como guas parta la segurdad en la manubucción, el usu calcaramente como quanta la materica, no educano comarse como parteritar la calcaramente a material se parteritor parteritor y pared no se action con calcarquiera tora materials e on encionada y pared no se action con calcarquiera tora materials e on encionada y que y parteritar parteritar en la tanto.
SECCION 16: OTKA INFORMACION	Fin de la ficha de d	Fin de la ficha de datos de seguridad
ACR12514	ACR12614	

				ł
			PREVENTION	
0		-783-9	ACUTE HAZARDS	
CAS #: 7727-37-9	UN #: 1066	EC Number: 231-783-9		

ICSC: 1198 March 1999

ICSC 1198 - NITROGEN (COMPRESSED GAS)

NITROGEN (COMPRESSED GAS)

22/6/2020

	ACUTE HAZARDS	PREVENTION	FIRE FIGHTING
FIRE & EXPLOSION	Not combustible. Heating will cause rise in pressure with risk of bursting.		In case of fire in the surroundings, use appropriate extinguishing media. In case of fire: keep cyfinder cool by spraying with water.

	SYMPTOMS	PREVENTION	FIRST AID
Inhalation Unc	Unconsciousness. Weakness. Suffocation. See Notes.	Use ventilation.	Fresh air, rest. Artificial respiration may be needed. Refer for medical attention.
Skin			
Eyes			
Ingestion			

CLASSIFICATION & LABELLING	According to UN GHS Criteria		Transportation	UN Classification UN Hazard Class: 2.2	e on behalf of ILO and WHO.
SPILLAGE DISPOSAL	Ventilation. Personal protection: self-contained breathing apparatus.	STORAGE	Fireproof if in building. Cool. Keep in a well-ventilated room.	PACKAGING	Provide the control of the cont

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NITROGEN (COMPRESSED GAS)	ICSC: 1198
PHYSICAL & CHEMICAL INFORMATION	CAL INFORMATION
Physical State: Appearance Odourless colourless compressed GAS.	Formula: N ₂ Molecular mass: 28.01 Bellinn noint-1987C
Physical dangers	Metting point: -210°C Solubility in water: poor
Chemical dangers	Relative vapour density (air = 1): 0.97
EXPOSURE & HEALTH EFFECTS	ALTH EFFECTS
Routes of exposure The substance can be absorbed into the body by inhelation. Effects of short-term exposure	Inhabition risk On loss of containment this substance can cause sufficiation by loweing the oxygen content of the air in confined areas. See Notes. Effects of long-term or repeated exposure
OCCUPATIONAL EXPOSURE LIMITS	XPOSURE LIMITS
ENVIRONMENT	NMENT
NOTES	ES
High concentrations in the air cause a deficiency of oxygen with the risk of unconsciousness or death. Check oxygen content before entering area.	e risk of unconsciousness or death.
ADDITIONAL INFORMATION	VFORMATION
EC Classification	

NITROGEN

1/2

2/2

228/020 (Sec 106) - Stuccon Cellellic (non-flenous) (Sec 106) - Stuccon Cellellic (non-flenous) (Sec 106) (Sec 106)

	ACUTE HAZARDS	PREVENTION	FIRE FIGHTING
FIRE & Ni	4ot combustible.		In case of fire in the surroundings, use appropriate extinguishing media.

	SYMPTOMS	PREVENTION	FIRST AID
Inhalation Cough.	Cough.	Avoid inhalation of dust.	Fresh air, rest.
Skin			
Eyes	Redness. Pain.	Wear safety goggles.	First rinse with plenty of water for several minutes (remove contact lenses if easily possible), then refer for medical attention.
Ingestion		Do not eat, drink, or smoke during work.	

CLASSIFICATION & LABELLING	According to UN GHS Criteria	Tenenorfation	UN Classification	on behalf of ILO and WHO.
SPILLAGE DISPOSAL	Personal protection: particulate filter respirator adapted to the airborne concentration of the substance. Sweep splited substance into covered containers.	STORAGE	PACKAGING	Constraints Constrain

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	ICSC: 1061
PHYSICAL & CHE	PHYSICAL & CHEMICAL INFORMATION
Physical State: Apparance Prellow-To-Greek-TO-BLACK CRYSTALS, DEPENDING ON PURITY Physical dangers	Formula: SIC Molecular ansa: 401 Subination point: 2000 Dentatio: 23 point Subility in water: none
Chemical dangers	
EXPOSURE &	L EXPOSURE & HEALTH EFFECTS
Routes of exposure	Inhalation risk A nuisence-causing concentration of airborne particles can be risadhed quicky when disporsed.
Effects of short-term exposure May cause mechanical infration.	Effects of long-term or repeated exposure
OCCUPATIONA	OCCUPATIONAL EXPOSURE LIMITS
TLV: (inhalable fraction): 10 mg/m ² , as TWA. TLV: (respirable fraction): 3 mg/m ³ , as TWA. MAK: carcinogen category: 2	
ENVI	environment
	NOTES
Thesi card does not spoy to allicon carded in "Through" or "Whistian" form. Thesi is oridone of fittoriss in mused dust shattory. It an risk of which is anhanced in perions with Manculosis Throus is no volcance alta slattor carbido (non-fittorus) suuces fittoris in the attention of other dust.	ker" form. of which is enhanced in persons with tuberculosis. fibrosis in the absence of other dusts.
ADDITIONA	ADDITIONAL INFORMATION

2/2

https://www.ilo.org/dyn/icsc/showcard.display?p_card_id=1061&p_edit=&p_version=2&p_lang=en

VALERIC ACID	ICSC: 0346
Pentanoic acid	
Propylacetic acid	
1-Butanecarboxylic acid	Mdy 2002
n-Pentanoic acid	
CAS #: 109-52-4	
UN #: 3265	
EC Number: 203-677-2	

	ACUTE HAZARDS	PREVENTION	FIRE FIGHTING
FIRE & EXPLOSION	FIRE & Combustible. Above 88°C explosive INO open fiames. Above 86°C use a Use water spray, powder, foam, EXPLOSION vapour/air mixtures may be formed. Dosed system and ventilation.	NO open flames. Above 86°C use a closed system and ventilation.	Use water spray, powder, foam, carbon dioxide.
	AVOID ALL CONTACT	AVOID ALL CONTACT! IN ALL CASES CONSULT A DOCTOR!	OCTORI
	SYMPTOMS	PREVENTION	FIRST AID
Inhalation	Burning sensation. Cough. Sore throat.	Use ventilation, local exhaust or breathing protection.	Fresh air, rest. Refer for medical attention.

	STINFLOWS	L'REVENTION	LIKSI AID
Inhalation	Burning sensation. Cough. Sore throat.	Use ventilation, local exhaust or breathing protection.	Fresh air, rest. Refer for medical attention.
Skin	Redness. Pain. Skin burns.	Protective gloves. Protective clothing.	Remove contaminated clothes. Rinse skin with plenty of water or shower. Refer for medical attention .
Eyes	Redness. Pain. Severe burns.	Wear face shield.	First rinse with plenty of water for several minutes (remove contact lenses if easily possible), then refer for medical attention.
Ingestion	Burning sensation. Abdominal pain. Do not eat, drink, or smoke during Shock or collapse.	Do not eat, drink, or smoke during work.	Rinse mouth. Give one or two glasses of water to drink. Do NOT induce vomiting. Refer for medical attention .

CLASSIFICATION & LABELLING	According to UN GHS Criteria	Transmontation	UN Classification	UN Hazard Class: 8	on behalt of ILO and WHO.
SPILLAGE DISPOSAL	Do NOT let this chemical enter the environment. Collect leaking liquid in covered containers. Wash away remainder with plently of water.	STORAGE	Separated from strong bases.	PACKAGING	Property by an international group of experts of behalf of LO and WiO and Neuron ansettanoo of the European Commean. Proceeding of the European Commean.

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EC Classification Symbol: C; R: 34-52/53; S: (1/2)-26-36:45-61

VALERIC ACID	ICSC: 0346
	PHYSICAL & CHEMICAL INFORMATION
Physical Stars, Appendince ColORIESS (JUGIO WITH CHAROCTERISTIC ODOUR. Physical damages The vacor is a break track Chemical damages The audutions is a weak acid.	Formula CgHing2, CHI(CHI),000H Moscular mices (CT Mosting mices (CT Mosting mices (CT Mosting mices (CT Mosting mices (CT) Mosting mices (CT) Most
EXPOSURE & H	EXPOSURE & HEALTH EFFECTS
Routes of exposure Routes of exposure impactor. The automore and by advanced into the body by inhaletion and by impactor. The automore and advances in the eyers, siten and respiratory tract. Correlate on ingestion.	Inhalation risk. Inhalation risk. Non ordension can be given stoot the risk at which is harmful concernation of this substance in the art is reached on exponention at 20°C. Effects of long-term or repeated exposure
OCCUPATIONAL	OCCUPATIONAL EXPOSURE LIMITS
ENVIRG	ENVIRONMENT
The substance is harmful to aquatic organisms.	
NO	NOTES
ADDITIONAL	ADDITIONAL INFORMATION

VALERIC ACID

2/2

1/2

APPENDIX 3: CHROMATOGRAPH MODE



Model 1 (30 °C ramp)							
Columns:							
Flow	mL/min ²	mL/min		Hold (min)			
Initial		1.	.0	0.00			
Ramp1	0.0	0.0		0.00			
Ramp2	0.0	0.0		0.00			
Post				0.00			
	Set point						
Pressure	2.272 bar						
Flow	1.0 mL/min						
Average Velocity	25 cm/sec						
Inlets:							
Successful and Marcal							
Mode split							
On/Off		Set point					
On	Heater	200 °C					
On	Pressure	2.272 bar]				
On	Total Flow	103 mL/min]				
			-				
Split Ratio	100.0/1	Split Flow	99.9 mL/min				
			_				
On/Off							
On	GasSaver	15.0 mL/min	2.00 min				

Not changeable



Model 2 (25 °C ramp)								
Columns:								
Flow	mL/min ²	mL/min		Hold (min)				
Initial			1.0	0.00				
Ramp1	0.0		0.0	0.00				
Ramp2	0.0		0.0	0.00				
Post				0.00				
	Set point			-				
Pressure	2.272 bar							
Flow	1.0 mL/min							
Average Velocity	25 cm/sec							
Inlets:								
Mode split								
On/Off		Set point						
On	Heater	200 °C						
On	Pressure	2.272 bar						
On	Total Flow	103 mL/min						
			_					
Split Ratio	100.0/1	Split Flow	99.9 mL/min]				
On/Off								
On	GasSaver	15.0 mL/min	2.00 min]				

Not changeable

APPENDIX 4: CHROMATOGRAMS

<u>Chromatogram 1</u>: 1mL (5mL H₂0 + 1mL valeric acid + 1mL GVL + 1mL LA) + some acetone wastes. To determinate H₂0 using Model 1. Manual injection.

<u>Chromatogram 2</u>: 1mL (5mL H₂0 + 1mL valeric acid + 1mL GVL + 1mL LA) + 2mL GVL. To determinate GVL using Model 1. Manual injection.

<u>Chromatogram 3</u>: 1mL (5mL H₂0 + 1mL valeric acid + 1mL GVL + 1mL LA) + 2mL LA. To determinate LA using Model 1. Manual injection.

<u>Chromatogram 4</u>: 1mL (5mL H₂0 + 1mL valeric acid + 1mL GVL + 1mL LA) + 2mL valeric acid. To determinate valeric acid using Model 1. Manual injection.

<u>Chromatogram 5</u>: H₂O + Acetone in bigger proportion. To determinate Acetone using Model 1. Manual injection.

<u>Chromatogram 6</u>: 1mL (5mL H_20 + 1mL valeric acid + 1mL GVL + 1mL LA) using Model 2. We changed the model, so since we know in which order each peak appears, we can determine their retention times with this Model. Manual injection.

<u>Chromatogram 7</u>: 0,5mL H₂0 +0,5mL AL To determinate AL using Model 2. Manual injection.

<u>Chromatogram 8</u>: Analysis to check if SiC was inert. Manual injection.

Chromatogram 9: In this chromatogram GVL was detected but not clearly. Direct injection.

Chromatogram 10: In this chromatogram GVL was detected. Manual injection.

CHROMATOGRAM 1: RETENTION TIME DETERMINATION

Data File C:\HPCHEM\1\DATA\PROVES\DEFAULTF.D

Sample Name: prova_00



*** End of Report ***

CHROMATOGRAM 2: RETENTION TIME DETERMINATION

Data File C:\HPCHEM\1\DATA\PROVES\DEFAULTF.D



*** End of Report ***

Sample Name: prova 00

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CHROMATOGRAM 3: RETENTION TIME DETERMINATION

Data File C:\HPCHEM\1\DATA\PROVES\DEFAULTF.D





*** End of Report ***

CHROMATOGRAM 4: RETENTION TIME DETERMINATION

Data File C:\HPCHEM\1\DATA\PROVES\DEFAULTF.D



Sample Name: prova 00

CHROMATOGRAM 5: RETENTION TIME DETERMINATION

Data File C:\HPCHEM\1\DATA\PROVES\DEFAULTF.D

Sample Name: prova_00



*** End of Report ***

CHROMATOGRAM 6: RETENTION TIME DETERMINATION

Data File C:\HPCHEM\1\DATA\PROVES\DEFAULTF.D



6890 2/4/2020 7:17:14 PM Hazael

CHROMATOGRAM 7: RETENTION TIME DETERMINATION

Data File C:\HPCHEM\1\DATA\PROVES\DEFAULTF.D

Sample Name: prova_00



*** End of Report ***

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CHROMATOGRAM 8: RETENTION TIME DETERMINATION

Data File C:\HPCHEM\1\DATA\PROVES\DEFAULTF.D



*** End of Report ***

Sample Name: prova 00

CHROMATOGRAM 9: GVL NOT CLEARLY DETECTED

Data File C:\HPCHEM\1\DATA\PROVES\DEFAULTF.D





CHROMATOGRAM 10: GVL DETECTED

Data File C:\HPCHEM\1\DATA\PROVES\DEFAULTF.D



*** End of Report ***

Sample Name: prova 00