Combining Internet Monitoring Processes, Packaging and Isotopic
 Analyses to Determine The Market Structure: Example of Gamma
 Butyrolactone

4

# 5 Abstract

The Internet is becoming more and more popular among drug users. The use of websites and 6 forums to obtain illicit drugs and relevant information about the means of consumption is a 7 growing phenomenon mainly for new synthetic drugs. Gamma ButyroLactone (GBL), a 8 9 chemical precursor of Gamma Hydroxy Butyric acid (GHB), is used as a "club drug" and also in drug facilitated sexual assaults. Its market takes place mainly on the Internet through online 10 websites but the structure of the market remains unknown. This research aims to combine 11 digital, physical and chemical information to help understand the distribution routes and the 12 structure of the GBL market. Based on an Internet monitoring process, thirty-nine websites 13 14 selling GBL, mainly in the Netherlands, were detected between January 2010 and December 2011. Seventeen websites were categorized into six groups based on digital traces (e.g. IP 15 addresses and contact information). In parallel, twenty-five bulk GBL specimens were 16 purchased from sixteen websites for packaging comparisons and carbon isotopic 17 measurements. Packaging information showed a high correlation with digital data confirming 18 the links previously established whereas chemical information revealed undetected links and 19 provided complementary information. Indeed, while digital and packaging data give relevant 20 information about the retailers, the supply routes and the distribution close to the consumer, 21 the carbon isotopic data provides upstream information about the production level and in 22 particular the synthesis pathways and the chemical precursors. A three-level structured market 23 has been thereby identified with a production level mainly located in China and in Germany, 24

an online distribution level mainly hosted in the Netherlands and the customers who order onthe Internet.

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28 Keywords: Internet monitoring processes; Packaging; Carbon isotopic analyses; Gamma

29 Butyrolactone; Intelligence; Market structure; Digital Trace; Drug Profiling

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# 31 **1. Introduction**

Due to the availability of many new recreational drugs online, the Internet is a key source of 32 information in identifying new trends of drug abuse. Despite efforts of different governments 33 to combat this virtual market, the online purchase of psychoactive compounds, in particular 34 35 synthetic drugs, has grown significantly. In fact, due to the magnitude and accessibility of the Internet, the amount of customers buying substances called "legal highs" or "research 36 chemicals" has increased [1]. Online platforms are generally hosted in countries where these 37 substances are still permitted. This new trend tends to replace traditional exchanges between 38 consumers and dealers on the street. Moreover, due to the rapid spread of new synthetic 39 substances, the precise effects are still unknown or little known [2]. This study has been 40 focused on Gamma Butyrolactone (GBL), the main chemical precursor of Gamma Hydroxy 41 Butyric acid (GHB), which is directly converted into GHB in the body after ingestion [3, 4]. 42 43 Recent surveys indicate that the consumption of GBL is a growing trend among drug users aiming to replace the consumption of GHB. This seems to be mainly due to its availability on 44 the Internet and the lack of control in many countries [5]. The global production of GBL 45 worldwide exceeds 200,000 tons per year and as it is an important and common industrial 46 solvent which is used in large quantities as an intermediate in the synthesis of plastics, 47

48 polymers or pesticides among others, it is hardly controlled under national or international49 laws [6].

Nevertheless, the use of the Internet opens new perspectives to analyze and understand these 50 phenomena in order to implement more proactive strategies against trafficking. In that 51 respect, some projects have emerged in Europe under the umbrella of the European 52 Monitoring Centre for Drugs and Drug Addiction (EMCDDA) using Internet monitoring 53 processes and dedicated databases storing newly identified substances in order to give crucial 54 information regarding health risks. However, little research has been published concerning 55 Internet monitoring processes of known psychoactive substances aimed at acquiring a better 56 understanding of the market for intelligence purposes [7]. 57

From a forensic perspective, such global and strategic knowledge is fundamental in order to 58 understand the research problem, select a representative sampling and make relevant and 59 realistic assumptions. When it comes to common source inference or chemical profiling, these 60 assumptions will depend on manufacturing processes such as syntheses pathways, the number 61 of batches or the amount of psychoactive substance contained in batches as well as supply 62 63 routes and distribution networks. Therefore, the knowledge of the market structure from the production line to the consumer is essential to interpret chemical links between two or more 64 specimens. Such links are assumed to depict that specimens are probably coming from a same 65 "source" but how can we define this specific "source"? [8, 9] Concerning GBL and depending 66 on the market structure, the "source" could be defined as the producer, the synthesis pathway, 67 the batch or even the Internet retailer. 68

In the present project, we investigate the potential of gathering several forensic information namely, digital, physical and chemical to gain a better understanding of the overall GBL market structure. An Internet monitoring methodology has been implemented for the

- 3 -

detection and the following of websites selling GBL and the detection of links based on digital traces. From specimens purchased on different websites, packaging information was collected in order to confirm or detect new links. Finally, by comparing the variation in the carbon isotopic composition of GBL specimens, chemical linkages were identified. The combination of digital, physical and chemical links is then globally discussed.

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# 78 2. Materials and methods

#### 79 2.1. GBL Internet Monitoring Process

A monitoring methodology of the websites selling GBL was set up. The aim of this 80 monitoring process was to establish an effective search strategy of the GBL websites and 81 82 above all to detect similarities during the creation and the hosting of the websites in order to infer linkages between these entities [10, 11]. For that purpose, twelve specific keywords 83 combining different GBL names "GBL", "Gamma Butyrolactone" or "y-Butyrolactone" with 84 simple keywords in English like "cleaner", "buy" or "supplier" were initially chosen for the 85 search of GBL websites with Google.com® search engine. Complementary searches were 86 carried out with other keywords ("purchase/sell/order") and GBL street names using different 87 tools like natural language ("Where to buy Gamma Butyrolactone?") and Google® 88 multilingual search. 89

As a second step, an automatic alert system (Google Alert®) and a page change monitoring system (ChangeDetection.com) were implemented in order to detect the emergence of new websites and provide information about those already identified. Both alert systems were used to follow the evolution and the dynamic of the online GBL market. The results obtained by this monitoring process were then compared to previous results collected in 2010 [12].

#### 96 2.1.1 Websites geolocation

Once the websites were indexed, several elements were gathered in order to carry out 97 geographical analyses. Initially, information concerning the provenance of the product, and 98 the distribution worldwide such as the geographical area of sale were considered. On one 99 hand, the study of website content was performed, including the visible content and the 100 language used to comment source code, the product origin (sometimes written on the website) 101 and the contact address. On the other hand, analysis of the technical data such as extracting 102 the IP address with the WHOIS protocol gives an indication of the hosting country, the 103 country of the registrar, the registrant and the technical contact (Table 1). 104

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**Table 1:** Information collected for website geolocation

Geographical information	Information collected
Origin and distribution	Provenance of the product and geographical area of sale
Website content	Site language, source code language and contact address
Technical data	Geolocation of the IP address, registrar and registrant country
	and technical contact

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## 109 2.1.2 Digital links

In addition to geographical information, other similarities between the GBL websites were sought. For that purpose, various information was collected and compared between the different websites:

# Website content: URL addresses, logos on the browser tab (i.e. favicon), contact data (name, address, phone number) and bank details.

Technical data: IP addresses of the server hosting the site, the subnets of the IP address and the contact of the owner, the HTTP header of the server, Google®
markers whose "Google Adsense®" and "Google Analytics®", scripts on the source

118 code, the "robot.txt" file and finally the WHOIS data of the domain name (registrar119 and registrant information).

All these searches were performed manually, using "nslookup" to identify the IP addresses and the "Firebug" plugin of Firefox® to read the scripts. Subnets information and the contact of the owner were obtained on the "www.iana.org" website while the data related to the registration of the domain name and the HTTP header information were obtained on the sites "whois.domaintools.com" and "www.webrankinfo.com/outils/header.php" respectively.

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#### 126 *2.2. Sampling*

Twenty-five bulk GBL specimens were purchased online from sixteen websites among the thirty-six detected in order to proceed with physical and chemical analyses. Multiple specimens were also ordered from a subset of the websites (**Table 2**). It was decided to order new specimens every six months assuming that after such a period, a whole batch may

- 131 probably have been sold.
- **Table 2:** Bulk GBL specimens ordered on the websites.

GBL Websites (URL)	Names and Specimens
www.alco-international.nl	Alco International
www.all-chemicals.com	All-chemicals
www.alloycleaner.com	Alloycleaner
www.clean-crystal.com	GBLCleanCrystal
www.cleanmagic.de	Cleanmagic
www.cleanmpower.com	CleanMPower
www.cleanstar24.pl	Cleanstar 24 (Specimen 1)
www.cleanstar24.pl	Cleanstar 24 (Specimen 2)
www.gammabutyrolactone.cn	Everchem
www.gbl24.com	GBL24 (Specimen 1)
www.gbl24.com	GBL24 (Specimen 2)
www.gbl24.com	GBL24 (Specimen 3)
www.gblcleaner.eu/.nl	GBLcleaner
www.gblstarcleaner.com	GBLstarcleaner (Specimen 1)
www.gblstarcleaner.com	GBLstarcleaner (Specimen 2)
www.gblstarcleaner.com	GBLstarcleaner (Specimen 3)
www.liquidsoap.cc	Liquidsoap
www.multicleaner.eu	Multicleaner (Specimen 1)

www.multicleaner.eu	Multicleaner (Specimen 2)
www.multicleaner.eu	Multicleaner (Specimen 3)
www.odegasupercleaner.nl	Odergasupercleaner (Specimen 1)
www.odegasupercleaner.nl	Odergasupercleaner (Specimen 2)
www.rapidcleaner.com	Rapidcleaner
www.shineandbright.com	Shine & Bright (Specimen 1)
www.shineandbright.com	Shine & Bright (Specimen 2)

Eight GBL specimens coming from four different alleged producers were also purchased on a trade website (www.alibaba.com) selling products directly from manufacturers. These specimens originated in China, one of the most important GBL producers. No digital analyses were performed on this website since it gathers together products from several producers.

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The twenty-five bulk GBL specimens were received in different plastic bottles. Physical information, such as the color, size, diameter and type of stopper, was collected from the bottle. Additionally, information including the name, size, design and language, was obtained from the label. All this information was used for comparisons between specimens and to identify "physical links". No physical or analytical analyses were performed on bottles, only visual comparisons.

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# 148 2.4. Chemicals and Carbon isotopic measurements on a GC/C/IRMS

Dichloromethane (≥ 99.9%) was purchased from Merck (Darmstadt, Germany). ε-Caprolactone (≥ 99.0%, Lot 1256826) was obtained from Fluka (Buchs, Switzerland). Helium (Quality 60, > 99.9999%) and carbon dioxide gas (Quality 40, > 99.99%) were purchased from Carbagas (Domdidier, Switzerland). Tetradecanoic acid methyl ester (C14:0, #14M,  $C_{15}H_{30}O_2$ ,  $\delta^{13}C = -29.98 \pm 0.02\%$ , > 99.0%) was obtained from Arndt Schimmelmann (Indiana University, Department of Geological Sciences, Biogeochemical Laboratories, 1001 East 10th Street, Bloomington, IN, USA).

<sup>140 2.3.</sup> Physical links: Packaging

Carbon isotopic measurements were performed three times at six months interval. The carbon 157 isotope measurements were performed on a Delta V Plus IRMS system (ThermoFisher 158 Scientific Inc., Bremen, Germany) coupled to a Trace GC Ultra Gas Chromatograph via a 159 Finnigan<sup>™</sup> GC-C/TC III interface (ThermoFisher Scientific Inc., Bremen, Germany). The 160 samples were injected via a TriPlus<sup>TM</sup> autosampler (ThermoFisher Scientific Inc., Bremen, 161 Germany). The mass spectrometer consisted of an electron impact source held at 3.0 kV 162 acceleration voltage for CO<sub>2</sub> gas, a magnet and three Faraday collectors for measurement of 163 the ions at m/z 44, 45 and 46. Concerning the sample preparation, 150 µL of  $\varepsilon$ -caprolactone 164 (Internal Standard, 20 µg/mL in dichloromethane) and 50 µL of tetradecanoic acid methyl 165 ester (Isotope Calibrator, 10 µg/mL in dichloromethane) were mixed to 100 µl of GBL (100 166 µg/mL in dichloromethane). After vortex-mixing for 5 sec, GC/C/IRMS analyses were 167 performed according to the methodology previously published [12]. 168

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The symbol  $\delta$  is the standard notation for expressing carbon isotope ratios. It is defined as parts per thousand deviations of isotopic compositions from that of Vienna Pee Dee Belemnite (VPDB) and is calculated according to [13]:

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$$\delta^{13}C / \%_{0} = \frac{({}^{13}C/{}^{12}C)_{sample} - ({}^{13}C/{}^{12}C)_{standard}}{({}^{13}C/{}^{12}C)_{standard}} \times 1000$$
(1)

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Acquisition and evaluation of the GC/C/IRMS data were performed with the ISODAT 2.5
software (ThermoFisher Scientific, Bremen, Germany).

#### 179 2.3.2 Threshold setting and chemical links

The intra variability values were calculated as the differences in the carbon isotope ratio 180 between each of the replicates of the twenty-five GBL bulk. The inter variability values were 181 established as the differences between the carbon isotope ratio means of the sixteen different 182 GBL retailers. The evaluation of the overlapping area between the inter variability and the 183 intra variability of the distribution  $\delta^{13}$ C -values was performed by calculating the true and 184 false positive rates. This study has been carried out for operational and intelligence purposes 185 in order to increase our understanding of the GBL market and provide strategic information 186 instead of providing evidence in court. For that reason, the threshold has been set up to detect 187 all possible links (true positive rate maximized) and accepting higher values for the false 188 positive rate. On the contrary, if the aim were to compare GBL specimens for evidence 189 purposes, the false positive rate would have been minimized at the expense of the true 190 positive rate. Therefore, a threshold of  $\delta^{13}$ C 0.9‰ was established enabling discrimination 191 between "chemically" linked and "non-chemically" linked GBL specimens. The statistical 192 analyses and calculations were performed with Excel® while I2 Analyst's Notebook® was 193 used for the visual representation of the linkages. 194

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#### 196 **3. Results and Discussion**

#### 197 3.1 Internet Monitoring Process

A total of thirty-six websites were initially detected with our methodology in June 2011. Twenty-seven of these websites were found using the keyword "GBL cleaner" while only four were found by complementary keyword combinations or natural language search and two Ukrainian websites were detected with the multilingual search of Google®. From June to December 2011, the follow-up of the GBL websites showed three additional sites. Finally, thirty-nine websites selling GBL were discovered with the automatic alert systems previouslyestablished.

These results were compared with a previous search performed in January 2010 as part of another study in which twenty-six websites were found using a traditional search on Google.com® without any alert or automatic systems (**Figure 1**) [12].



As shown in Figure 1, sixteen new websites were detected and six were closed from January 2010 to June 2011. Only three new websites were detected and eight closed from June to December 2011. Moreover, twenty websites, already identified in January 2010, were detected in June 2011 and twenty-seven websites, already brought out in June 2011, were found in December 2011.

These results showed that the GBL online market seems fairly stable between 2010 and 2011 and how easy it is to find GBL websites with simple keywords and without any previous knowledge of the market. Some of the new websites detected between 2010 and 2011 were probably due to the improvement of specific search criteria and the establishment of the automatic alert system. In fact, a high number of websites (69%) remain from previous searches and only a small number have been closed. On one hand, this stability can be a relevant indicator of the lack of control measures against these websites on the Internet. On the other hand, the websites are versatile and may tend to be located in countries where the substance is not under control and where the laws are more flexible. Then, research was undertaken to identify the countries that supply the GBL and the ones who host the websites.

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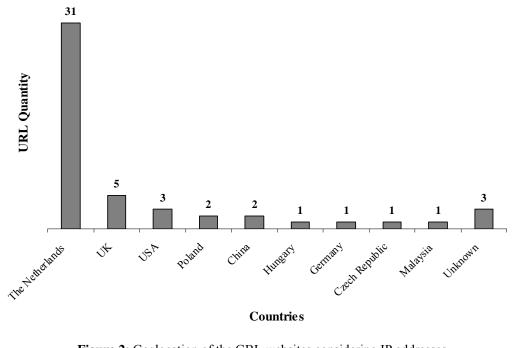
#### 227 3.1.1 Websites geolocation

Most of the websites distribute GBL worldwide with some restrictions for countries such as 228 USA, Canada, Australia, New Zealand and Norway where GBL is prohibited. Some of the 229 websites (less than 50%) indicate that the provenance of the GBL is mainly China and 230 231 Germany. Indeed, GBL manufacturers are mostly based in China, Germany (BASF), India (Balaji Group) or the USA (Lyondell and Ashland) and belong to important chemical 232 companies [14]. In 2012, BASF manufactured more than 60,000 tons per year while in China, 233 more than 20 GBL producers were active in 2005 and the total capacity of GBL 234 manufacturing was reported in 2006 to be around 50,000 tons per year, 33,000 of which were 235 exported [15]. 236

One of the issues was to investigate if the websites were hosted in the manufacturing 237 countries. It is worth noting that IP addresses may not be directly linked to the retailer's 238 location. Indeed, websites may be hosted in foreign countries and spoofing strategies may 239 also be used to hide the true location of the machine. Therefore, Information found on 240 websites including the source code language, the site language and the personal information 241 242 (contact address, phone number, etc.) has also been investigated for comparison. The combination of these pieces of information is highly relevant, in particular the source code 243 and site language, which are mainly related to the designer of the website who may certainly 244

use his own language for commenting the source code. By comparing the geolocation using Information found on websites and IP addresses geolocation, the most significant difference concerns three websites that had IP addresses in Czech Republic, Germany and the Netherlands but were built in polish and the contact addresses on these websites were from Poland.

According to the results shown in Figure 2 and detailed in Table 3, more than 60 % of the 250 websites were coming from the Netherlands where GBL is not controlled by national law. 251 These results point out that most of the websites are hosted in countries that do not have 252 official manufacturing companies such as the United Kingdom, Poland and mostly the 253 Netherlands. Since the geolocation of websites is different from GBL producer countries, 254 255 these GBL Internet websites seem to act as retailers between costumers and manufacturing companies. They have an intermediary level function, which will be called "the distribution 256 level" in the market structure. 257



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Figure 2: Geolocation of the GBL websites considering IP addresses

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263	Table 3: Results of the websites geolocation	n. DNS	(Data Not Shown)	1

Nomes and anosimons	Sussimons	Geolocation	Geolocation	
Names and specimens	Specimens	(Technical info)	(Websites content)	
Alco International	Specimen 1	The Netherlands	The Netherlands	
All-chemicals	Specimen 1	Germany	Poland	
Alloycleaner	Specimen 1	DNS	UK	
GBLCleanCrystal	Specimen 1	Bulgaria	Poland	
Cleanmagic	Specimen 1	The Netherlands	The Netherlands	
CleanMPower	Specimen 1	Poland	Poland	
Cleanstar 24 (Spec. 1)	Specimen 1	The Netherlands	Poland	
Cleanstar 24 (Spec. 2)	Specimen 2	The Netherlands	Poland	
Everchem	Specimen 1	China	China	
GBL24 (Spec. 1)	Specimen 1	DNS	Poland	
GBL24 (Spec. 2)	Specimen 2	DNS	Poland	
GBL24 (Spec. 3)	Specimen 3	DNS	Poland	
GBLcleaner	Specimen 1	The Netherlands	DNS	
GBLstarcleaner (Spec. 1)	Specimen 1	The Netherlands	Germany	
GBLstarcleaner (Spec. 2)	Specimen 2	The Netherlands	Germany	
GBLstarcleaner (Spec. 3)	Specimen 3	The Netherlands	Germany	
Liquidsoap	Specimen 1	DNS	UK	
Multicleaner (Spec. 1)	Specimen 1	The Netherlands	The Netherlands	
Multicleaner (Spec. 2)	Specimen 2	The Netherlands	The Netherlands	
Multicleaner (Spec. 3)	Specimen 3	The Netherlands	The Netherlands	
Odergasupercleaner (Spec. 1)	Specimen 1	USA	The Netherlands	
Odergasupercleaner (Spec. 2)	Specimen 2	USA	The Netherlands	
Rapidcleaner	Specimen 1	Czech Republic	Poland	
Shine & Bright (Spec. 1)	Specimen 1	The Netherlands	The Netherlands	
Shine & Bright (Spec. 2)	Specimen 2	The Netherlands	The Netherlands	

264

265 Complementary research has been undertaken to highlight similarities between various 266 websites in order to make assumptions about the amount of retailers and how they are 267 structured.

#### 268 3.1.2 Digital links

Seventeen of the thirty-nine websites were categorized into six groups (**Figure 3**). Most of the websites were linked by at least two separate sources of information. For instance, in the upper right section of Figure 3, "Magic Cleaner" and "Astro Lab" were connected by three pieces of information: the IP address, contact information on the website and the *Google*® *Analytics* markers. These markers are unique codes specific to customers and are used by webmasters to follow the traffic on their websites. Therefore, this code is relevant to linking websites handled by the same group of persons. Moreover, phone numbers and IP addresses confirm the highlighted similarities and reinforce the linkages established between these websites.

It is important to note that online GBL retailers do not seem to be articulated around a small number of persons. The structure of the organization includes many small entities (one or two websites), which seem to be independent of each other, except for the two groups containing three and six websites.

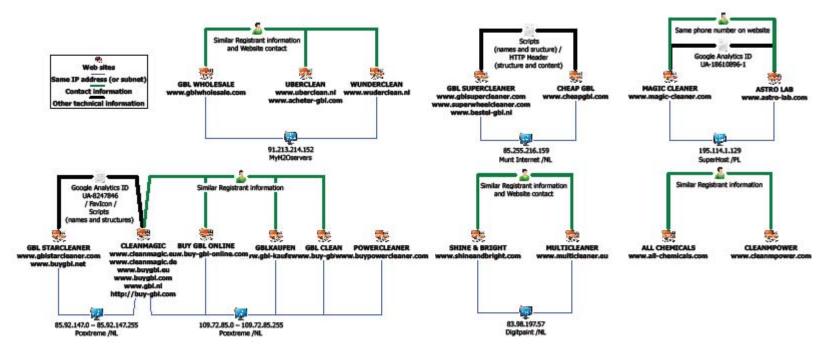


Figure 3: Schema showing all the GBL websites linked by digital information

As previously highlighted, the digital information concerns the distribution level and little information has been obtained regarding the production level and the dynamism of the market (interactions and flows between retailers and manufacturers). For instance, do retailers tend to order always from the same company or do they change their producer contacts to find cheaper products or on the contrary, purer products? To answer these questions, the investigation of the physical and chemical information is required.

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## 297 *3.2 Physical links: Packaging*

Packaging information may provide clues on the retailers and the supply routes based on the 298 hypothesis that the retailers receive the GBL in big containers and have to pack the samples in 299 bottles before sending them with specific labels. Indeed, the results (Table 4) show that 300 packaging tends towards confirming the links previously established by the digital 301 information and seems to help understand the distribution network. Sixteen specimens (ten 302 websites) were linked and categorized into five groups. Three of the five groups were already 303 connected by digital information while two groups of two websites were only linked by 304 physical information. 305

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### **Table 4:** Results of the obtained physical links

Groups	Physical link	Type of link	Specimens linked
1	А	Bottle	GBLstarcleaner (Specimen 1), GBLstarcleaner (Specimen 2),
			GBLstarcleaner (Specimen 3) and Cleanmagic
2	В	Label	Liquidsoap and Alloycleaner
3	С	Label	Shine & Bright (Specimen 1) and Multicleaner (Specimen 1)
	D	Bottle	Shine & Bright (Specimen 1), Multicleaner (Specimen 1) and
			Multicleaner (Specimen 2)
	Е	Bottle	Shine & Bright (Specimen 2) and Multicleaner (Specimen 3)
	F	Label	Shine & Bright (Specimen 2), Multicleaner (Specimen 2) and
			Multicleaner (Specimen 3)
4	G	Label	CleanMPower and All-chemicals

5	Н	Bottle	Cleanstar 24 (Specimen 1), Cleanstar 24 (Specimen 2) and
			Rapidcleaner

# 309 *3.3 Determination of the chemical classes*

Carbon isotopic analyses were performed on twenty-five GBL specimens coming from sixteen different websites as well as on eight GBL specimens coming from four alleged producers. The results showed a wide variation of  $\delta^{13}$ C-values ranging from -23.11‰ to -45.74‰. Concerning the alleged Chinese GBL producers, the carbon isotopic values ranged from -27.54‰ to -29.83‰. All the  $\delta^{13}$ C-values were associated with very low standard deviation (lower than 0.3‰) of triplicate analyses (**Table 5**).

First, it was decided to compare the  $\delta^{13}$ C-values with the geographical origin of GBL 316 specimens specified on the websites even if this information is not always reliable. The results 317 showed connections between the  $\delta^{13}$ C-values and the country of origin of the GBL specimens. 318 Indeed, GBL specimens originating from Germany showed carbon isotopic values ranging 319 from -45.74‰ (Cleanstar24 Specimen 1) to -41.70‰ (GBL24 Specimen 2) except for 320 GBL starcleaner Specimen 1 (-27.09‰) while the  $\delta^{13}$ C-values obtained for the Chinese 321 specimens ranged from -30.02‰ (Everchem Specimen 1) to -23.11‰ (Shine&Bright 322 Specimen 1). The enriched  $\delta^{13}$ C-value of GBL starcleaner Specimen 1 (-27.09‰) is probably 323 due to the fact that this website (GBL starcleaner), which sells two GBL qualities at different 324 prices (German quality is more expensive than the Chinese one), seeks to sell lower GBL 325 quality specimens from China under the label of a higher GBL quality from Germany. 326 Concerning Odegasupercleaner, the website sent us, as specimen 1, GBL coming either from 327 328 Germany or the USA without specifying which of the two and, as specimen 2, GBL supposedly from Poland that has no official manufacturing companies. Likewise, two 329 countries of origin were specified by the website Cleanmagic (China and USA; -37.50%). 330

331	Therefore, the country of origin given by these websites should be taken with precaution and
332	they were not categorized neither in the Chinese nor in the German groups. Through these
333	results, the potential of carbon isotopic analyses to differentiate between GBL originating
334	from Germany and China was demonstrated but more specimens of producers from Germany
335	and the USA are required as well as more information about the unknown specimens (DNS).
336	Moreover, the differences in the $\delta^{13}\text{C}\text{-values}$ of GBL between pairs of specimens from
337	different websites were calculated and the chemical classes were determined according to the

threshold of 0.9‰ (in the  $\delta^{13}$ C-values) previously established (Figure 4). This threshold 338 allowed us to highlight five different chemical classes from A to E and four specimens 339 (Cleanmagic, GBLcleaner, Alloycleaner and Shine & Bright) were not connected to any other 340

specimen analyzed (Table 5 and Figure 4). 341

Websites Names	Specimens	GBL origin	$\delta^{13}$ C-means (‰)	SD (‰)	Chemical class
Cleanstar 24	Specimen 1	DNS	-45.74	0.10	А
CleanMPower	Specimen 1	DNS	-45.52	0.05	А
GBL24	Specimen 1	Germany	-45.50	0.12	А
GBL24	Specimen 3	Germany	-44.27	0.21	В
Rapidcleaner	Specimen 1	Germany	-43.94	0.12	В
GBLCleanCrystal	Specimen 1	DNS	-43.60	0.02	В
All-chemicals	Specimen 1	Germany	-43.58	0.11	В
Cleanstar 24	Specimen 2	DNS	-43.58	0.16	В
GBL24	Specimen 2	Germany	-41.70	0.12	С
Odegasupercleaner	Specimen 1	Germany/USA	-41.55	0.11	С
Cleanmagic	Specimen 1	China/USA	-37.50	0.04	-
GBLcleaner	1	DNS	-32.06	0.08	-
Everchem		China	-30.02	0.09	D
Producer 1	Specimen 1	China	-29.83	0.07	D
Producer 1	-	China	-29.75	0.10	D
Alco International		China	-29.73	0.05	D
Multicleaner		DNS	-29.64	0.08	D
Producer 2	1	China	-29.45	0.04	D
Odegasupercleaner	-	Poland	-29.44	0.06	D
Producer 2	1	China	-29.18	0.10	D
Multicleaner	-	DNS	-29.13	0.12	D
Multicleaner	1	DNS	-29.05	0.05	D
Shine & Bright	1	China	-28.95	0.18	D
Producer 3		China	-27.99	0.11	Ε
Liquidsoap	-	DNS	-27.97	0.10	Е
GBLstarcleaner	1	China	-27.86	0.11	Е
Producer 4	1	China	-27.85	0.05	Ε
GBLstarcleaner	•	China	-27.82	0.03	Ē
Producer 4		China	-27.67	0.09	Ε

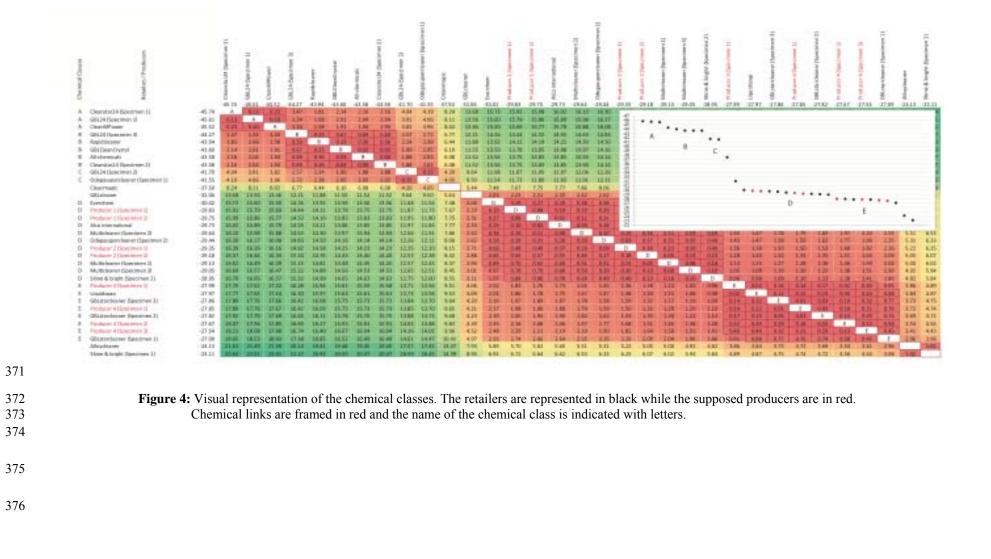
Table 5. Carbon isotonia values for CPL appairments ordered online DNS (Data Not Shown) 342

GBL starcleanerSpecimen 1Germany-27.090.18EAlloycleanerSpecimen 1DNS-24.130.08-Shine & BrightSpecimen 1China-23.110.04-	Producer 4	Specimen 3	China	-27.54	0.22	E
	GBLstarcleaner	Specimen 1	Germany	-27.09	0.18	Е
Shine & Bright Specimen 1 China -23 11 0.04 -	Alloycleaner	Specimen 1	DNS	-24.13	0.08	-
	Shine & Bright	Specimen 1	China	-23.11	0.04	-

In fact, classes A, B and C correspond to GBL specimens originating from Germany and classes D and E are related to specimens originating from China. Likewise, Chinese specimens of class D can be related with Chinese producers 1 and 2 while GBL specimens of class E correspond to Chinese producers 3 and 4.

348 Based on previous work, it can be assumed that the wide range of carbon isotopic values obtained for GBL specimens is due to the synthetic pathways and the starting material 349 employed. In fact, several chemical precursors may be potentially used for the manufacturing 350 351 of GBL at an industrial level [12]. The major portion of GBL is currently produced from the 352 dehydrogenation of 1,4 Butanediol (1,4-BD), which is manufactured via the REPPE process from the reaction of acetylene with formaldehyde [16]. Other GBL manufacturing processes 353 are based on economically attractive raw chemicals such as maleic anhydride [17, 18] and 354 dimethyl maleate [19] via the Davy process. Almost all companies manufacturing GBL in 355 Europe use the REPPE process in their production chain whereas in Asia both REPPE and 356 Davy processes are often used [14]. New eco-friendly manufacturing routes using the 357 biotransformation of natural starting materials such as glucose have also emerged recently 358 359 [20]. Therefore, it may be expected that specimens of classes A, B, C (German specimens) and classes D, E (Chinese specimens) were manufactured via different synthesis processes 360 and different feedstocks. Concerning the small differences in the  $\delta^{13}$ C-values between A, B 361 and C as well as between D and E, it may be hypothesized that these specimens were 362 manufactured through the same synthesis process but perhaps with different starting materials 363 or different batches of the same starting material. This could explain the small differences in 364 the  $\delta^{13}$ C-values found between these classes. Considering that the retailers can purchase GBL 365

- 366 from various producers, one of the assumptions to explain the differences in the  $\delta^{13}$ C-values
- 367 for specimens between class A and E may be that different GBL samples were mixed by the
- 368 retailers causing variation in the carbon isotopic value.



#### 377 *3.4 Combining digital, physical and chemical information*

By adding chemical information, nine new websites were indirectly connected (Figure 5). It 378 is worth emphasizing that the link between "Shine&Bright" and "Multicleaner", which was 379 already observed with digital information, was confirmed by physical and chemical 380 information. This example clearly demonstrates the potential of combining physical 381 observations with chemical information. In fact, by merely exploiting chemical information, 382 "Multicleaner specimens 1, 2 and 3" would have been linked to "Shine&Bright specimen 2" 383 and not to "Shine&Bright specimen 1". By adding physical observations performed on the 384 label and on the bottle, "Shine&Bright specimen 1" has been related to "Multicleaner 385 specimens 1 and 2". As highlighted previously, packaging takes place at the distribution level 386 387 and is therefore probably related to retailers more than producers. The combination of all of these sources of information strongly reinforces the hypothesis that the same group of people 388 handled these websites. Moreover, some specimens ordered on the same website were linked 389 to different websites. For instance, "Odegasupercleaner specimen 1" was connected to 390 "GBL24 specimen 2" but they were not linked to other specimens. Knowing that the 391 392 specimens were ordered every 6 months, it may be hypothesized that the manufacturers supplying these websites might have been replaced by producers selling cheaper or better 393 quality products. This example enhances the fact that the chemical information doesn't link a 394 395 specimen and its supplier (website) or two suppliers together. This information allows to link a specimen with a producer or to conclude that two unrelated websites got their supply from 396 the same producer. Likewise, it is worth noting that chemical information provides relevant 397 398 and complementary information regarding the production level and the flow routes between retailers and manufacturers. 399

400 In conclusion, it may be inferred that the online GBL market is structured in at least three levels. First, a production stage based on few synthesis routes and starting materials mainly 401 402 located in China and Germany. Then, an intermediary level of supply and distribution highly visible on the Internet through websites mainly hosted in the Netherlands has been brought up 403 and finally customers and consumers who order on the websites. In the industry, many official 404 companies handle pure GBL without manufacturing it. A previous study, in 2003, by the 405 Swedish National Institute of Public Health in relation to the flow of pure GBL in Sweden, 406 showed that companies handling GBL might be importers, distributors, users or a 407 combination of them [6]. All these trades are controlled but thefts cannot be excluded. The 408 results obtained during the current study show that the combination of various sources of 409 410 information opens new perspectives to understanding and obtaining background knowledge as well as providing relevant information in order to assist international organizations in their 411 fight against drug trafficking, not only for GBL but also for others substances. The 412 methodology proposed could be implemented in routine at two levels. The first level includes 413 all the digital information and, in particular, the monitoring of the websites that can be easily 414 415 standardized by national and international organizations (such as the federal police, EUROPOL or INTERPOL for instance). The second level covers all the physical and 416 chemical analyses that would be performed, depending on the casework, by specialized 417 418 laboratories. Finally, these laboratories should ideally convey information to national and international organizations. 419

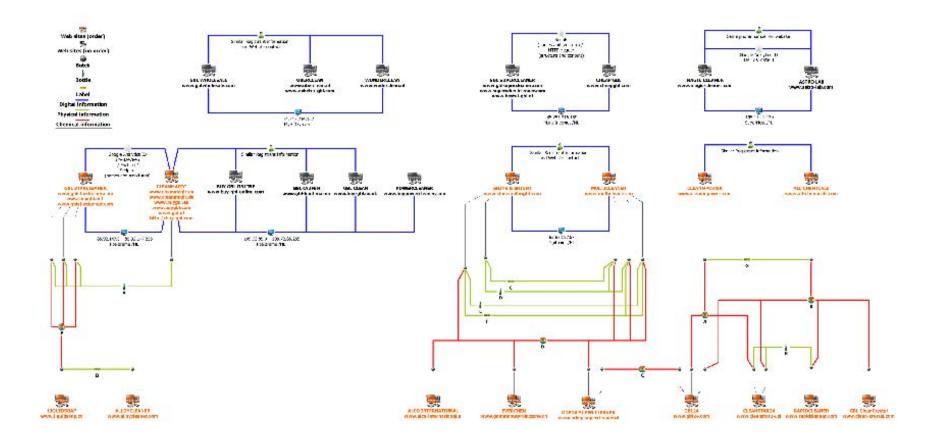


Figure 5: Schema combining digital, physical and chemical linkages obtained for the GBL websites.

#### 425 **4. Conclusion**

A methodology for the monitoring of GBL websites was developed. A simple detection strategy using twelve keyword combinations showed great results in regard to more complex web searches. This monitoring process also required the design of a dedicated system that accommodated and organized collected data in a fitted working memory in order to detect relationships between websites and therefore, provide relevant information about the distribution market.

The results obtained so far emphasize the feasibility of a forensic approach combining multiple sources of information. They demonstrate that digital and physical data provide relevant information about the retailers, the supply routes and the distribution while the carbon isotopic composition provides upstream information about chemical links and concerns mainly the production stage. In order to further study the production step, isotopic profiling of GBL samples manufactured in the same industrial plant should be undertaken to establish the carbon isotopic variation within and between different batches.

The combination of various sources of information opens new perspectives to exploit forensic information in order to improve the common knowledge of drug market structures, not only for GBL but also for other substances. This knowledge is of major importance to correctly interpret the notion of "source inference". Finally, this research shows how digital traces may be combined with physical and chemical profiles extracted from specimens to gain a better insight into the phenomena from intelligence and investigative perspectives at national and international levels.

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