

Ping, Li, and Fleming: The First Three Studies Where DMAA Was Found in Geranium

written by Mike Roberto | August 4, 2017

This is **part one** of a **six page** series researching whether DMAA (1,3 dimethylamylamine) is a natural constituent of geranium flowers. All six parts are linked from our main DMAA in Nature / Geranium page.

Note from Mike, Founder of PricePlow

Thanks for joining us. A tremendous amount of research went into these articles, and since there is so much written (over 13,000 words), it will be best absorbed in chunks. Take it one part at a time and enjoy.

29	2-Heptanamine, 5-methyl-	5-甲基-2-庚胺	C ₁₂ H ₂₇ N	129	0.23
30	2-Hexanamine, 4-methyl-	4-甲基-2-己胺	C ₁₁ H ₂₅ N	115	0.44
31	Isomer of 2-Heptanamine, 5-methyl-	3-甲基-2-己胺	C ₁₂ H ₂₇ N	129	0.29

Amount of 31 identified peaks (%) in total analysis based on peak area - % 07

**Part one of six:
The disputed
"Pro-DMAA Studies"**

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We'll start with the disputed studies, and why the government wanted more "research".

We'll briefly begin this research series with the three studies where DMAA was found *and* published as detected.

Many industry scientists, including the FDA's, have questioned these three papers, but we aren't going to dwell on them *too* much because the objections are indeed reasonable.

Instead, it's the research discussed in our *subsequent* parts where things start to get wildly interesting – and that's where we'll spent more time. But first, let's get warmed up and understand how it all began:

1. The 1996 Ping Study that Started it All: "A study on the chemical constituents of geranium oil"

Method of Measurement: GC-MS (Gas Chromatography-Mass Spectrometry)

The original study referenced when declaring that 1,3 Dimethylamylamine is found in nature, the **Ping study** (formally titled "A study on the chemical constituents of geranium oil") is what kicked off the entire DSHEA-compliance argument.

This study was published in Mandarin in the Journal of Guizhou Institute of Technology.

In this study, it is line 30 that is of interest, where 0.66% of the content was determined to be "2-Hexanamine, 4-methyl-", a synonym for DMAA / Methylhexaneamine / 1,3-Dimethylamylamine:

29	2-Heptanamine, 5-methyl-	5-甲基-2-庚胺	$C_8H_{19}N$	129	0.23
30	2-Hexanamine, 4-methyl-	4-甲基-2-己胺	$C_7H_{17}N$	115	0.66
31	Isomer of 2-Heptanamine, 5-methyl-	5-甲基-2-己胺	$C_8H_{19}N$	129	0.29
Amount of 31 identified peaks (%) in total analysis based on peak area					95.07

The Ping study was only published in Mandarin,[1] and original copies are incredibly difficult to find. So when it was referenced, many questioned the translations used. This translated source comes from Exhibit 53 in Hi-Tech's lawsuit against the FDA.[1]

The above image comes from the Hi-Tech court case, but as you can see, the PDF has been marked up. The *true* original study is available through one of USPLabs' response letters to the FDA:[2,3]

29	2-Heptanamine, 5-methyl-	5-甲基-2-庚胺	$C_8H_{19}N$	129	0.23
30	2-Hexanamide, 4-methyl-	4-甲基-2-己胺	$C_7H_{17}N$	115	0.66
31	Isomer of 2-Heptanamine, 5-methyl-	5-甲基-2-己胺	$C_8H_{19}N$	129	0.29
Total surface content which is currently determined – 95.07					
29	2-Heptanamine, 5-methyl-	5-甲基-2-庚胺	$C_8H_{19}N$	129	0.23
30	2-Hexanamide, 4-methyl-	4-甲基-2-己胺	$C_7H_{17}N$	115	0.66
31	2-Hexanamide, 5-methyl-	5-甲基-2-己胺	$C_7H_{17}N$	129	0.29
已定性的组分占色谱总流出峰面积的百分数					95.07

Provided in USPLabs' response letters to the FDA,[2,3] you can see how difficult it was to obtain a true source copy of this study. Was it *really* DMAA Ping found at 0.66%? The question is in 2-hexaneamide-4-methyl vs 2-hexanamine-4-methyl.

As you can see, a major issue with the Ping study is that the *original* translation to English identified it as 2-hexaneamide-4-methyl. Translation experts have argued that this was really supposed to be 2-hexanamine-4-methyl, which is in fact DMAA.

Despite the confusion, the Ping study itself was even confirmed by an FDA employee, Dr. Robert J. Moore, who stated in a 2010 email that DMAA could be

found in geranium oil which had “a fairly long history of food use as an essential oil”:[4]

From: Moore, Robert J [mailto:]
Sent: Wednesday, October 13, 2010 10:36 AM
To: Amy Eichner, Ph.D.
Subject: RE: NDI's

Dr. Eichner: I've inserted some comments below.

bob

From: Amy Eichner, Ph.D. [mailto:]
Sent: Wednesday, October 13, 2010 11:55 AM
To: Moore, Robert J
Subject: RE: NDI's

Hello Dr. Moore-

Thank you so much for your thoughtful considerations on this matter. Your descriptions below help me to understand dietary ingredients and NDIs. I have just a couple of remaining questions.

- Can you give me some examples of plants that contain methylhexaneamine?

It is found in the oil of many geraniums - principally *Pelargonium graveolens*, the oil of which has a fairly long history of food use as an essential oil.

Emails uncovered by Hi-Tech Pharma's lawsuit show that even the FDA's own expert, Robert Moore, claimed that DMAA is in Geranium thanks to the Ping study.[4]

If you have trouble understanding the above email, note that Moore took the USADA (US Anti-Doping Agency) employee's email and simply put his response directly *under* her question. We boxed it in red for you, but the source cited has the original copy.[4] This is an “inline response” as opposed to the “top quoted” emails that we normally do these days, and is simply a different form of email etiquette.

Objections to the Ping Study:

- It is not in native English and is subject to translation, and was indeed mistranslated at first
- It did not use the same detection technology as studies that did *not* find DMAA
- It is not from a well-recognized lab, university, or researcher
- Nobody (to our knowledge) has ever found or spoken to Ping!
- Other studies that detected DMAA in geranium did not find it in such high concentrations
- The FDA has suggested that the plant material was not properly authenticated[5]
- The FDA also states that the study did not include reports of the *quality*

of the matches, such as comparing to known reference standards.[5]

- Is an *oil* enough to confirm basis in nature? Wouldn't it better to find DMAA in the raw plant material itself?

It was clear that this study wouldn't be enough to keep Jack3d and OxyELITE Pro on the market, so USPLabs then funded the following two studies:

2. The 2012 Li Study (The "Intertek Study"): "Identification and Quantification of Dimethylamylamine in Geranium by Liquid Chromatography Tandem Mass Spectrometry."

Method of Measurement: HPLC-ESI-QQQ

Between 2010 and 2012, USPLabs attempted to make their case that DMAA came from geranium. They funded the Li study,[5] which attempted (and indeed *succeeded*) in finding 1,3 Dimethylamylamine in Geranium (*Pelargonium graveolens*) plants taken from various provinces in China, including the same one as the Ping study (Guizhou).

The results:

Levels of 1,3-DMAA (1) and 1,4-DMAA (2) in geranium (*Pelargonium graveolens*) plant and geranium oil from different sources.

Sample ID	Source	Date of collection	1,3-DMAA, ng/g	1,4-DMAA, ng/g
070611-0164 (plant)	Yunnan, China	June 9, 2011	13.60	3.56
072811-1026 (plant)	Jiangsu, China	June 9, 2011	165.0	35.30
072811-1027 (plant)	Guizhou, China	June 5, 2011	365.0	9.12
051911-0588 (oil)	Jiangxi, China	–	13271	220.0
042911-0988 (oil)	Jiangxi, China	–	167.0	Not detected
042911-0989 (oil)	Jiangxi, China	–	377.0	Not detected

Table 5 of the Li / Intertek Study show that DMAA was clearly found.[6] But the FDA still had objections and some conspiracy theories of its own.[5]

To confirm the results and measurement equipment, the researchers assayed spiked samples of geranium plants at four different levels and the determined that the measurements were taken valid at levels *below* what was detected in the plants above (as low as 5ng per gram of material). So the equipment easily worked at levels needed.

This is often known as the “Intertek Study” since it was analyzed at Intertek, one of the most prominent detection labs in the world. Nobody questions the *data*, but the *source material* instead came under fire.

Objections to the Li study:

- It was paid for by USPLabs, a company with great interest in detecting DMAA
- The FDA claims that “*the study does not provide all of the information necessary to demonstrate proper authentication*”, such as the date of collection nor the plant’s habitats and growing conditions.[5]
- The isomers of DMAA found in the plant material were present in equal ratio, which does not often happen naturally and was not explained to the FDA’s satisfaction. This led to accusations that it was synthetic DMAA that was detected.

3. The 2012 Fleming Study: “Analysis and Confirmation of 1,3-DMAA and 1,4-DMAA in Geranium Plants Using High Performance Liquid Chromatography with Tandem Mass Spectrometry at Ng/g Concentrations.”

Method of Measurement: HPLC-ESI-QQQ

In addition to the Li study, USPLabs also partially funded the Fleming study, which also detected DMAA in three different geranium samples.[7] This study was performed at the University of Memphis.

Table 5 from the study shows the detection of the three samples, with the strongest detection in the Changzhou region’s flowers:

Analysis set 3: optimized extraction protocol results of geranium summer samples from Kunming, Guiyang, and Changzhou.

	1,3-DMAA			1,4-DMAA		
	Sample (ng/g)	Spike level (µg/L)	Percent recovery (%)	Sample (ng/g)	Spike level (µg/L)	Percent recovery (%)
Kunming 3	<10 ± 6 [*]	15.0	68 ± 3	<8.2 ± 0.3 [*]	15.0	64 ± 2
		25.0	74 ± 6		25.0	75 ± 9
Guiyang 3	<10 ± 6 [*]	15.0	75 ± 4	<8.1 ± 0.2 [*]	15.0	78 ± 1
		25.0	81 ± 8		25.0	84 ± 6
Changzhou 3	68.8 ± 36.5	15.0	76 ± 13	118 ± 45	15.0	86 ± 4
		25.0	79 ± 13		25.0	77 ± 7

Fleming also found DMAA,[7] but one of the samples was the same as the Li study sample!

Objections to the Fleming study:

- USPLabs also paid for a portion of this study
- The FDA again claims that “*vital collection and identification information... was not provided in the study to demonstrate proper authentication*”. [5]
- One of the samples from the Changzhou province that had DMAA detected was the same one as the Li study above. The researchers called this a “multi-center” study, but the FDA does not consider this “verification” since it did not use a different analytical technique.
- The isomers of DMAA found in the plant material were again present in equal ratio, and not explained to the FDA’s liking.

Not enough solid evidence?

At the time in 2012, the above three studies were the three most major pieces of evidence for the pro-DMAA side. As they are not slam dunks in the FDA’s eyes due to the objections above, and did not “override” the studies where DMAA was published as “not detected”, the FDA then sent their infamous warning letters, [8] which effectively got most of the DMAA supplements removed from the market.

But now is when things start to get *really* weird...

Up next: The three studies where DMAA *wasn’t* found... or *WAS* it?

The next three parts of this series (parts 2, 3, and 4) will make you question every research paper you’ve ever read.

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You can also see the list of DMAA Supplements still on the market and stay tuned to Hi-Tech Pharma's DMAA Lawsuit and Appeal Against the FDA.

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45. *Angelo Lisi, N. Hasick, R. Kazlauskas, C. Goebel; "Studies of methylhexaneamine in supplements and geranium oil"; Drug Testing and Analysis; Volume 3, Issue 11-12; November-December 2011 ; Pages 873-876; https://onlinelibrary.wiley.com/doi/10.1002/dta.392/abstract (full-text available at https://www.docdroid.net and https://www.webcitation.org/6sFt9cZbM)*