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Deputy Minister.

LABORATORY
OF THE
INLAND REVENUE DEPARTMENT
OTTAWA, CANADA.

BULLETIN No. 154

OIL OF LEMON



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OTTAWA, May 22, 1908.

W. J. GERALD, Esq.,
Deputy Minister of Inland Revenue.

SIR,—I beg to hand you a report upon 45 samples of Oil of Lemon, collected in February of this year, through the various inspectoral districts of the Dominion.

The results of analysis do not enable me to arrive at well-defined conclusion regarding these samples. The characteristics of Oil of Lemon are so vaguely described in the pharmacopoeias, and its composition has been so imperfectly worked out, that there is much difficulty in interpreting the analytical numbers. The present report must be regarded as a contribution to the study of Oil of Lemon, rather than as an application of methods of detecting adulteration. Definite methods can only be arrived at by the study of data such as are now submitted.

I beg to recommend the publication of this report as Bulletin No. 154; and I may add that the work herein recorded has been performed by Mr. A. Lemoine.

I have the honour to be, Sir,

Your obedient servant,

A. MCGILL,

Chief Analyst.

MEMORANDUM.

Oil of lemon (*Oleum Limonis*) is thus defined by the British Pharmacopœia (Edition of 1898):—

Characters and Tests.—Pale yellow, with the fragrant odour of the lemon, and a warm bitterish aromatic taste. Specific gravity 0.857 to 0.860. It should rotate the plane of a ray of polarized light not less than 59° to the right in a tube 100 millimetres long; and if 100 volumes be fractionally distilled, the 10 volumes first collected should not produce a rotation differing by more than 2° from that produced by the original oil.

The Pharmacopœia of the United States (Seventh Decennial Revision, 1890) gives the following definition:—

A pale yellow, limpid liquid, having the fragrant odour of lemon, and an aromatic, somewhat bitterish taste.

Specific gravity: 0.857 to 0.860 at 15° C. (59° F.). Its optical rotation should not be less than 60° to the right in a 100 Mm. tube, and at a temperature of about 15° to 20° C. (59° to 68° F.).

Soluble in three times its volume of alcohol, the solution being neutral or slightly acid to litmus paper; also soluble, in all proportions, in absolute alcohol, carbon disulphide, or glacial acetic acid.

When kept for some time, the oil should not develop a terebinthinate odour or taste (absence of *oil of turpentine* or of *other oils consisting chiefly of pinene*).

The National Dispensatory (5th edition) defines the specific gravity as about 0.857 to 0.863 at 15° C., and fixes the initial boiling point at 160° C. Seven (7) volumes of alcohol is quoted as characteristic for complete solution.

Oil of lemon is largely (about 90 per cent) composed of terpenes, the chief of which is limonene (C₁₀H₁₆). W. A. Tilden found 76 per cent of citrene (= d-limonene), boiling at 176° C.; 6 per cent of cymene (1:4, methylisopropyl benzene) boiling at 175° C., and other hydrocarbons.

The value of oil of lemon, for flavouring purposes, is chiefly due to the presence of citral, an aldehyde which constitutes from 4 to 8 per cent of the oil. (Squire's Companion, p. 398). Citral boils at 224° to 228° C., and is concentrated in the higher fractions when the oil is distilled.

It is because of the large proportion of terpenes in oil of lemon, that flavouring extracts must be made with strong alcohol. When too little alcohol is employed, the oil remains undissolved. It is claimed, however, that a large proportion of the citral, which is the chief flavouring component, is dissolved by a weakly alcoholic liquid, and in this way a so-called "terpeneless lemon extract" is prepared. Since the chief item of expense in lemon flavouring extract, is the alcohol, there is a temptation to manufacturers to employ so-called "terpeneless lemon oil." In Bulletin No. 89 are recorded 27 samples of extract of lemon, made with alcohol varying from 92.36 per cent to as low as 20.99 per cent strength. The oil of lemon found in solution varied from 15.5 per cent to a mere trace. In Bulletin 114, are recorded 110 samples of extract of

lemon, of which only 13 samples were made with alcohol of above 80 per cent, while 38 samples contained less than 30 per cent of alcohol. The oil of lemon was above 5 per cent in only 5 samples, and below 1 per cent in 78 samples.

When oil of lemon is subjected to repeated washing with a dilute alcohol, the undissolved residue, as it becomes poorer in citral, loses its commercial value for flavouring purposes, becoming more and more nearly a mixture of terpenes only. It is much to be desired that a workable method of detecting washed or exhausted oil of lemon should be devised. Up to the present time it is necessary to rely upon odour and taste. The German Pharmacopœia gives the following method of procedure:—
“One drop of the oil triturated with sugar and agitated with 500 grammes of water, should impart to the latter the pure odour of lemon.”

A. MCGILL.

Date of Collection.	Nature of Sample.	No. of Sample.	Name and Address of Vendor.	Cost.		Name and Address of Manufacturer or Furnisher as given by the Vendor.
				Quantity.	Cents.	
1908.						

DISTRICT OF NOVA SCOTIA—

Feb. 13	Oil of Lemon...	33604	National Drug Co., Halifax, N.S.	3 oz..	45	G. Ecora, Italy.....
" 14	"	33605	Buckley Bros., Halifax, N.S.	3 " ..	60	"
" 26	"	33606	W. H. Stevens, Dartmouth, N.S.	3 " ..	75	National Drug Co., Halifax, N.S.

DISTRICT OF PRINCE EDWARD ISLAND—

Feb. 17	Oil of Lemon...	31222	Johnson & Johnson, Charlotte- town.	3 oz..	60	Not known.....
" 18	"	31223	A. W. P. Gourlie, Summerside	3 " ..	60	Giacomo Ecora, Messina, Italy.
" 19	"	31224	Edgar Kier, Kensington.....	3 " ..	45	W. G. Polson & Co., King- ston, Ont.

DISTRICT OF NEW BRUNSWICK—

Feb. 12	Oil of Lemon...	29641	National Drug & Chem. Co., Ltd., Mill St., St. John, N.B.	3 oz..	75	Vendors
" 18	"	29642	E. Clinton Brown, cor. Union and Waterloo Sts., St. John, N.B.	3 " ..	90	G. E. Peirce, Messina, Italy.
" 21	"	29643	G. O. Spencer, Main St., Moncton, N.B.	3 " ..	75	The Can. Drug Co., Ltd., St. John, N.B.

DISTRICT OF QUEBEC—

Feb. 12	Oil of Lemon...	26392	E. O. Cloutier, Rivière du Loup en bas.	6 oz..	1 50	Polson Co., Kingston.....
" 18	Superior "	26463	Jos. Masson, 808 St. Valier, Quebec.	6 " ..	1 50	Polson, Kingston.
" 18	"	26464	" " " "	6 " ..	90	"

DISTRICT OF St. HYACINTHE—

Feb. 18	Oil of Lemon...	27948	F. F. Ansell, Sherbrooke.....	2½ " ..	65	Not known.....
" 21	"	27949	Dr. John West, Magog.	2½ " ..	90	"
" 22	"	27950	Dr. J. B. Comeau, Farnham..	3 " ..	75	Lyman, Sons & Co., Montreal

OIL OF LEMON.

Inspector's Report.	RESULTS OF ANALYSIS.					Appearance.	Remarks and opinion of the Chief Analyst.
	Index of Refraction.	Polarization.	Specific gravity.	Solubility in water, Alcohol.	Distills at		
	p. c.	p. c.	p. c.		p. c.		

R. J. WAUGH, INSPECTOR.

.....	5.79	+ 113	0.860	Soluble...	166°c	Light green. Limpid	
Sample taken from copper vessel, labelled Messina Oil.	60.3	+ 139	0.858	" ..	170.5°c	" "	
.....	59.5	+144.6	0.858	" ..	171°c	" "	

T. MOORE, INSPECTOR.

Could not remember from whom purchased; had been in stock for a long time.	62.8	+ 70.2	0.888	Soluble...	163°c	Very light green. Limpid.	Specific gravity is high; odour resembled turpentine.
.....	60.5	+ 64.2	0.860	Insoluble.	168.5°c	Yellow. Limpid.	
.....	62.5	+170.8	0.858	Soluble...	172.5°c	Light green.	

J. C. FERGUSON, INSPECTOR.

Imported in original 1 lb. coppers from Spain. Bottled by vendors.	57.9	+167.0	0.859	Soluble...	170°c	Yellowish green.	
From original copper package.....	60.2	+166.0	0.837	" ..	170°c	"	
.....	56.9	+ 80.0	0.860	" ..	167°c	Pale green. Limpid.	

E. BELAND, INSPECTOR.

.....	61.7	+158.3	0.864	Soluble...	172°c	Pale green. Limpid.	
.....	60.6	+ 170	0.856	" ..	170.5°c	" "	
.....	59.8	+ 65	0.803	Insoluble.	166°c	Yellow. Limpid.	

J. C. ROULEAU, INSPECTOR.

The balance in a 4 oz. bottle labeled "O.L. Lemons."	60.2	+132.4	0.859	Soluble...	166°c	Light green. Limpid	
" " " " " "	58.6	+ 106	0.871	" ..	166°c	" "	
Bottle labeled "O.L. Lemons."	59.4	+ 160	0.802	" ..	164°c	Yellow. Limpid.	

Date of Collection.	Nature of Sample.	No. of Sample.	Name and Address of Vendor.	Cost.		Name and Address of Manufacturer or Furnisher as given by the Vendor.
				Quantity.	Cents.	
1908.						
DISTRICT OF MONTREAL—						
Feb. 14	Oil of Lemon...	32574	E. F. Emery, 607 Notre Dame West, Montreal.	3 oz.	60	National Drug & Chem. Co., Montreal.
" 24	"	32575	Quenneville & Guerin, 700 St. Catherine St., Montreal.	3 "	75	Not known.....
" 24	"	32576	R. McNichol, 542 St. Catherine East, Montreal.	3 "	75	"
DISTRICT OF OTTAWA—						
Feb. 22	Oil of Lemon...	34118	National Drug & Chemical Co., Ottawa.	3 oz.	75	Not known.....
" 21	"	34119	The Ottawa Drug Co., Ottawa.	3 "	60	Giacomio Ecora & Co., Messina, Sicily.
" 17	"	34120	J. A. Plaunt, Renfrew.....	3 " 1 20	20	The Drug Trading Co., Toronto, Ont.
DISTRICT OF KINGSTON—						
Feb. 19	Oil of Lemon...	33064	W. W. Gibson, King street, Kingston.	3 oz.	39	H. Skinner & Co., Kingston.
" 10	"	33065	H. Wade, King st., Kingston.	3 "	60	N. C. Polson, Kingston....
" 10	"	33066	A. P. Chown, Princess street, Kingston.	3 "	60	Lyman & Son, Montreal....
DISTRICT OF TORONTO—						
Feb. 24	Oil of Lemon...	35039	John Mack, 1 James st., North, Hamilton.	3 oz.	75	Hamilton Coffee & Spice Co., Hamilton.
" 27	"	35031	H. Southcott, 99 St. Paul st., St. Catharines.	3 "	75	Lyman Bros. Co., Limited, Toronto.
" 28	"	35032	W. W. Ker, Cor. Main and Ferry sts., Niagara Falls South.	3 "	75	Giovanni Pasquale, Messina, Sicily.
DISTRICT OF LONDON—						
Feb. 14	Oil of Lemon...	30473	John Roberts & Co., druggist, Seaforth.	3 oz.	60	London Drug Co., London, Ont.
" 21	"	30477	W. S. R. Holmes, druggist, Clinton.	3 "	69	Drug Trading Co., Toronto..
" 29	"	30489	John Livingston, druggist, Listowell.	3 "	60	Elliot & Co., Wholesale druggists, Toronto.

OIL OF LEMON.

Inspector's Report.	RESULTS OF ANALYSIS.					Appearance.	Remarks and opinion of the Chief Analyst.
	Index of Refraction.	Polarization.	Specific gravity.	Solubility in 3 vols. Alcohol.	Distillat.		
	p. c.	p. c.	p. c.		p. c.		

J. J. COSTIGAN, INSPECTOR.

.....	61.7	+104.8	0.868	Insoluble.	167°c	Pale yellow. Limpid	
.....	58.3	+127.2	0.858	Soluble...	168.5°c	" "	
.....	61.7	+84.0	0.872	" ..	165°c	" "	

J. A. RICKEY, INSPECTOR.

Taken from 1 lb. copper container labeled, Best quality Oil of Lemon, hand pressed, specially imported, Nat. Drug & Chem. Co., Ltd., Montreal.	59.1	+160.6	0.862	Soluble..	170.5°c.	Very light green. Limpid.	
Imported by Vendors...	61.4	+38.4	0.865	Insoluble.	164°c.	Yellow. Limpid..	Polarization low.
Tin marked Oil of Lemon, super hand pressed, G. E. Peirce, Messina, Italy.	61.2	+150	0.867	Soluble..	168.5°c.	Colourless. Limpid.	

J. HOGAN, INSPECTOR.

.....	61.4	+165	0.860	Soluble..	169°c.	Light green. Turbid.	
.....	58.9	+165.4	0.858	" ..	171°c.	Light green. Limpid.	
.....	60.5	+167.4	0.858	" ..	171°c.	Light green. Limpid.	

H. J. DAGER, INSPECTOR.

Sample taken from behind dispensing counter.	57.4	+173.6	0.854	Soluble..	171°c.	Light green. Limpid.	
Sample taken from bottle in dispensing room.	59.1	+170.8	0.857	" ..	171°c.	Colourless. Limpid.	
Sample taken from original copper and labeled Strictly pure.	60.6	+169	0.857	" ..	171°c.	Yellow. Turbid ..	

T. KIDD, INSPECTOR.

.....	60.9	+160.0	0.859	Soluble..	169°c.	Very light green. Limpid.	
.....	60.0	+165.0	0.858	" ..	170.5°c.	" " ..	
.....	60.5	+162.0	0.860	" ..	171°c.	" " ..	

Date of Collection.	Nature of Sample.	No. of Sample.	Name and Address of Vendor.	Cost.		Name and Address of Manufacturer or Furnisher as given by the Vendor.
				Quantity.	Cents.	
1508.						

DISTRICT OF WINDSOR—

Feb. 18	Oil of Lemon . . .	34502	H. O. Fleming	3 oz. . .	75	National Drug Co.
" 20	"	34518	Anderson & Nelles, London. .	3 " . .	60	Dodge & Olcott, New York. .
Apl. 9	"	34527	T. H. Duncombe, St. Thomas. .	3 " . .	30	Nat. Drug Co., London.

DISTRICT OF MANITOBA—

Feb. 19	Oil of Lemon . . .	25874	Elmwood Drug Store, Elmwood P.O., Winnipeg.	3 oz. . .	1 20	The Bole Drug Co., Winnipeg, Man.
" 24	"	25875	Barkers Drug Store, Glenboro, Man.	3 " . .	75
" 28	"	25876	Sanders Drug Store, Carman, Man.	3 " . .	75	Martin Bole Wynne Co., Winnipeg, Man.

DISTRICT OF CALGARY—

Feb. 21	Oil of Lemon . . .	28889	Bole Drug Co., Calgary.	3 oz. . .	60	Unknown
" 22	"	28890	Oliver Bros., Calgary.	3 " . .	60	Evans & Sons, Ltd., Montreal.
" 22	"	28891	W. Maclean, Calgary.	3 " . .	60	Nat. Drug Co., Montreal.

DISTRICT OF VANCOUVER—

Feb. 17	Oil of Lemon . . .	34224	H. McDowell & Co., Corner Granville and Hastings sts., Vancouver, B.C.	3 oz. . .	75	J. B. Horner, New York.
" 17	"	34225	Leslie Henderson, Cor. Granville and Georgia sts., Vancouver, B.C.	4 " . .	1 50	" "
" 17	"	34226	Le Patourel & McRae, Granville st., Vancouver, B.C.	3 " . .	1 50	Lyman Sons & Co., Montreal

DISTRICT OF VICTORIA—

Feb. 28	Oil of Lemon . . .	34864	D. E. Campbell, Victoria, B.C.	3 oz. . .	75	Bush, London, Eng.
" 28	"	34868	Wm. Jackson & Co., Victoria, B.C.	3 " . .	75	Burgoyne, Burbidges & Co., London, Eng.
" 28	"	34871	Geo. A. Fraser, Victoria, B.C.	3 " . .	75	" "

OIL OF LEMON.

Inspector's Report.	RESULTS OF ANALYSIS.					Appearance.	Remarks, and opinion of the Chief Analyst.
	Index of Refraction.	Polarization.	Specific gravity.	Solubility in 3 vols. Alcohol.	Distillate		
	p. c.	p. c.	p. c.				

J. TALBOT, INSPECTOR.

.....	58.0	+164.2	0.858	Soluble.	169°c.	Colourless. Limpid.
.....	65.6	+154.2	0.878	" ..	170°c.	Very light green. Limpid.
.....	61.2	+132.0	0.861	" ..	169°c.	Colourless. Limpid.

A. C. LARIVIERE, INSPECTOR.

.....	61.4	+162.0	0.862	Soluble.	170°c.	Colourless. Limpid.
Bottle labelled The Pulford Leonard Drug Co., Winnipeg. Would not testify for purity.	61.7	+160.0	0.865	Insoluble.	166°c.	Light green. Limpid.
.....	59.5	+161.4	0.858	Soluble.	170.5°c.	" "

R. W. FLETCHER, INSPECTOR.

.....	59.8	+162.4	0.859	Soluble.	170.5°c.	Pale green. Turbid.
.....	58.3	+160.4	0.858	" ..	171°c.	" Limpid.
.....	60.5	+164.0	0.858	" ..	170.5°c.	" " ..

J. F. POWER, INSPECTOR.

Superior hand pressed; guaranteed.	61.5	+160.0	0.857	Soluble.	171.5°c.	Very light green. Limpid.
.....	60.2	+168	0.857	" ..	171°c.	" "
.....	49.8	+150.4	0.858	" ..	115°c.	Yellowish green....

D. O. SULLIVAN, INSPECTOR.

.....	65.1	+154.4	0.860	Soluble.	171°c.	Greenish. Limpid.
.....	60.2	+160.0	0.858	" ..	170.5°c.	" "
.....	61.2	+161.4	0.858	" ..	171.5°c.	" "