assumption is made, only nitrogen values need be obtained for most samples.

Silver complexing should be applicable to new oilseeds although recognition of the end point requires some practice to achieve the desired accuracy. This analytical procedure could become further complicated if color bodies unique to a certain oilseed are present. In addition, the stability of thioglucosides in boiling water is not known and may differ according to type. A short initial boiling period is necessary in the thioglucoside isolation step of this method to inactivate enzymes, but boiling should not continue beyond 5 min to avoid a breakdown of thioglucosides.

The water-extraction method is also subject to error if thioglucosides are broken down during extraction. In its application to oilseeds other than crambe, the presence of water-soluble sulfur compounds other than thioglucosides is a source of error. Such compounds include water-soluble peptides or proteins containing significant quantities of sulfur amino acids. As with the sulfate ion method, the solution should be thoroughly boiled to insolubilize the protein.

With these precautions in mind, the methods outlined should be useful in determining the thioglucoside content of members of the Cruciferae. The sulfate ion and silver complex methods are particularly well suited to screening programs where a large number and variety of thioglucosides may be encountered.

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Oil and Protein Content, and Oil Composition of the Seeds of Some Plants of the Canadian Prairies

E. C. M. COXWORTH, Saskatchewan Research Council, Saskatoon, Saskatchewan

Abstract

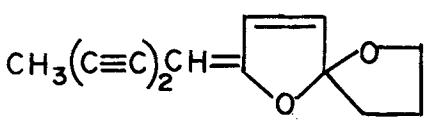
The oil and protein content are reported for the seeds of 19 plant species selected for their possible crop potential for the Canadian prairie region. Data on seed oil composition are reported for the 12 species which contained greater than 15% seed oil.

Introduction

THE SEEDS OF A NUMBER of plant species, mostly uncultivated plants of the Canadian prairie region, were investigated for oil and protein content. The composition of the oil was also determined for species with seeds containing more than 15% oil. Only species which were believed to have relatively high yields of seed, which held their seed well and which appeared reasonably easy to harvest, were examined. The combination of high yields of seeds, high content of useful oils in the seeds and recognizable hardiness in the plants are attributes which should enhance the potential for field crop consideration and possible development. Familiarity with the composition of the oils and with the oil content of wild plant seeds might lead also to by-product utilization of such seeds separated from more orthodox seed production during harvesting and cleaning operations.

Previous investigations, notably those of Earle and his associates (1) (2), have included data on many plant species of the Canadian prairie region. The present investigation has sought to supplement the literature with respect to species not previously studied, as well as to contribute information where data on particular species appeared incomplete.

Of the 19 plant species investigated, 12 were found to contain sufficient seed oil to warrant examination of its composition. As judged by the spectroscopic and GLC retention data, the major fatty acids of most of these oils were the usual C_{16}, C_{18} and C_{20} fatty acids, and only small amounts of epoxy, hydroxy or conjugated dienoid fatty acids were detected. The seed oil of *Hackelia americanum* (Boraginaceae family) contained substantial amounts of the $\Delta^{6,9,12}C_{18}$ trienoic and the $\Delta^{6,9,12,15}C_{18}$ tetraenoic fatty acids. These have already been reported by Craig and Bhatty (4) and by Kleiman et al. (5) to be present in the seed oil of many species of the Boraginaceae family. Both the seed and the other above ground parts of *Artemisia biennis* were found to contain significant amounts of the heterocyclic polyyne I, tentatively identified on the basis of spectroscopic evidence. This particular polyyne has already been isolated from two *Matricaria* species by Bohlmann and his associates (6).



I

Materials and Methods

Seeds of *Artemisia biennis, Hackelia americanum, Atriplex hortensis, Axyris amaranthoides* and *Rumex fennicus* were obtained from plants collected near Saskatoon during the fall and winter of 1963 and the fall of 1964. Seeds of *Gypsophila paniculata, Saponaria vaccaria, Chenopodium rubrum, Chenopodium hybridum* var. *gigantospermum, Galeopsis tetrahit, Moldavica parviflora* (*Dracocephalum parviflorum*), and *Lappula echinata* were supplied from the weed seed collection, Plant Ecology Department, University of Saskatchewan. The seeds of all the other species investigated were supplied by commercial seed houses. In the case of *Coreopsis tinctoria,* commercial seed

TABLE I

Analytical Data on Seeds

Family	Species	Common name	Component analyzed: Plant part	wt/1000 (g.)	% Oil (d.b.)	% Protein (d.b.)
Polygonaceae	*Rumex fennicus*		Achene	1.1	4	20.8
Chenopodiaceae	*Chenopodium rubrum*	Red goosefoot	Calyx plus pericarp and seed	0.5	2	16.6
	Chenopodium hybridum var. *gigantospermum*	Maple-leaved goosefoot	Pericarp and seed	1.3	7	9.8
	Atriplex hortensis		Pericarp and seed	8.8	6	31.3
	Axyris amaranthoides	Russian pigweed	Pericarp and seed	1.4	7	18.6
Caryophyllaceae	*Gypsophila paniculata*	Baby's breath	Seed	0.7	6	15.8
	Saponaria vaccaria	Cow cockle	Seed	3.9	4	14.2
Ranunculaceae	*Delphinium ajacis*	Larkspur	Seed	1.3	29	24.0
Loasaceae	*Mentzelia lindleyi*		Seed	0.7	33	26.5
Boraginaceae	*Hackelia americanum*	Nodding stickseed	Nutlets	1.0	32	13.3
	Lappula echinata	Blue bur	Nutlets	2.0	26	16.2
Labiatae	*Galeopsis tetrahit*	Hemp nettle	Nutlets	1.6	30	19.2
	Moldavica parviflora	American dragonhead	Nutlets	2.3	20	14.0
Cucurbitaceae	*Echinocystis lobata*	Wild cucumber	Seed minus seed coat	130	41	42.3
Campanulaceae	*Campanula rapunculoides*		Seed	0.25	28	21.5
	Platycodon grandiflora	Balloon flower	Seed	0.9	34	29.4
Compositae	*Artemisia biennis*	Biennial-wormwood	Achene	0.07	28	21.6
	Coreopsis drummondii		Achene	1.4	17	16.0
	Coreopsis tinctoria	Common tickseed	Achene	0.2	24	24.4

was grown, and the flowering plant identified to confirm the identity of the seed. Other commercial seed was accepted as being correct as to species without further confirmation.

The dried seed were ground and then extracted with hot n-hexane, either in a Butt-type extractor or in a Soxhlet extractor modified to give a constant drain of hot solvent from the extractor. Both types of apparatus gave similar yields of oil. Nitrogen was determined by a procedure (7) similar to the AOCS method (8).

Analysis of the Seed Oil of Those Species Having Greater than 15% Seed Oil

Iodine values, refractive indices, polyunsaturated fatty acids and unsaponifiables were determined by AOCS procedures (8). Epoxide content was measured by the method of Morris and Holman (9). Saponification equivalents were obtained by the procedure of Van Etten (10). Iodine values from refractive indices were calculated using the regression equation obtained by Earle and his associates (15); *Artemisia biennis* oil and *Mentzelia Lindleyi* (No. 1 sample) oil gave high I_2 values compared to Wijs I_2 values, all other I_2 values from refractive indices being quite close ($\pm$ 8 I_2 units) to Wijs I_2 values.

The oils were converted into the methyl esters either by interesterification, using sodium methoxide as a catalyst (11), or by saponification of the oil, extraction of the unsaponifiables with ethyl ether, acidification of the extracted soaps, and conversion of the free fatty acids into methyl esters by use of methanol containing boron trifluoride-etherate as catalyst (4). The saponification followed by acid catalyzed esterification technique was only used on those oils having a high percentage free fatty acids, i.e. *Artemisia biennis* and *Delphinium ajacis*. The methyl esters of *Artemisia biennis* oil were also prepared by washing the oil with diluted sodium carbonate and then carrying out the interesterification with sodium methoxide in methanol on the neutral (dried) portion of the oil. In general the base catalyzed interesterification was preferred since it was expected to avoid major decomposition of methyl esters of any epoxy or hydroxy fatty acids which might be present (12) (13).

For the GLC analysis of the methyl esters, a conventional commercial apparatus employing flame ionization detection was used. For separation by chain length, a 6 ft $\times$ 0.25 in. copper column containing 5% silicone grease on Haloport F was employed, operated at 225C and 60 ml/min helium. For separation of individual methyl esters, a 6 ft $\times$ 0.25 in. column contaniing 12% diethyleneglycol succinate on Anachrom A (60–70 mesh) was used at 205C and 25 ml/min helium. To confirm the relative amounts of 9,12,15-linolenic and eicosenoic acids, the methyl

TABLE II

Analytical Data on Seed Oils

Species	Saponification equivalent	% Unsaponifiables	Epoxides as % epoxy-oleic	IR spectrum of oil	Conjugated acids (as % C_{18}) diene[a]	R.I. at 40C	I_2 value (Wijs)	I_2 value from GLC
Delphinium ajacis	301	1.9	0.0	High free fatty acid	0.1	1.4627	98	97
Mentzelia lindleyi No.1[e]	302	1.5	1.8	Weak bands at 3500, 1600,978 cm^{-1}	4.3	1.4691	126	126
No.2[f]	—	—	—	Usual	—	1.4680	129	129
Hackelia americanum No. 1[e]	296	1.3	0.0	Usual	0.3	1.4718	172	
No. 2[f]	—	—	—	Usual	—	1.4730	178	176
Lappula echinata	293	1.0	0.5	Usual	0.4	1.4769	212	209
Galeopsis tetrahit	293	0.9	0.8	Usual	0.7	1.4713	161	158
Moldavica parviflora	303	—	0.0	Usual	0.4	1.4743	192	195
Echinocystis lobata No. 1[e]	310	4.2	0.0	Usual	1.6	1.4673	132	135
No. 2[f]	—	—	—	Usual	—	1.4687	135	137
Campanula rapunculoides	298	1.5	0.0	Usual	0.1	1.4702	162	162
Platycodon grandiflora	301	1.7	0.2	Usual	0.4	1.4680	140	139
Artemisia biennis	295	5.3	0.8[b]	Many bands 2250–800 $cm.^{-1}$ high free fatty acid[c]	polyyne interferes	1.4719	141[d]	143
Coreopsis drummondii	292	—	0.4	Usual	1.7	1.4685	135	135
Coreopsis tinctoria	302	2.2	0.3	Usual	1.2	1.4686	142	144

[a] = no conjugated trienoic acids were detected
[b] = methanol solubles removed
[c] = by titration 8% free fatty acid
[d] = on methyl esters after removal unsaponifiables
[e] = 1963 seed sample
[f] = 1964 seed sample

TABLE III

Fatty Acid Composition (GLC Results) of Seed Oils.

Species	Polyester column results									Silicone column results			
	16:0 %	16:1 %	18:0 %	18:1 %	18:2 %	18:3 %	Other fatty acids %			16 %	18 %	20 %	other %
Delphinium ajacis	3.3	0.5	0.9	59.9	14.2	1.4	20:0 = 0.1	20:1=18.5	20:2=0.7	3.4	77.7	18.8	
Mentzelia lindleyi No. 1[e]	11.2		3.0	20.3	56.8	1.3	14:0 = 0.2	Polar=7.2		11.8	81.0		Polar=7.2
No. 2[f]	10.4	0.3	2.7	18.8	63.0	1.8	14:0 = 0.2	Polar=2.8		10.6	86.5		14=0.1 Polar=2.8
Hackelia americanum No. 2[f]	6.8	0.3	1.5	20.6	17.5	19.3	18:3[a]=12.4	18:4=10.5	20:1=4.4	7.8	81.8	4.9	22=4.9
							20:2 = 0.2	22:0= 0.2	22:1=4.8				24=0.6
							24:1 = 1.3	20:0= 0.2					
Lappula echinata	6.4	0.4	1.9	14.1	15.2	33.6	18:3[a]= 8.1	18:4=17.0	20:1=2.7	6.6	90.2	3.0	22=0.2
							22:0 = 0.1	22:1= 0.5					
Galeopsis tetrahit	4.8		0.9	27.0	45.0	22.1	20:0 = 0.3			4.7	94.9	0.3	
Moldavica parviflora	5.8	0.8	2.6	7.8	28.7	53.3	14:0 = 0.3	20:0= 0.7		6.1	92.9	0.7	14=0.3
Echinocystis lobata No. 1[e]	10.3		3.4	14.6	69.0	1.5	14:0 = 0.3	20:0= 0.6	22:0=0.3	10.8	88.6	0.6	
No. 2[f]	9.5	0.2	3.7	13.3	70.1	1.7	14:0 = 0.1	20:0= 0.8	22:0=0.6	9.6	90.3	0.1	
Campanula rapunculoides	5.7	0.2	2.9	9.2	67.8	14.1	14:0 = 0.1			6.2	93.7		14=0.1
Platycodon grandiflora	7.3		3.3	15.5	73.0		20:0 = 0.9			7.6	91.7	0.7	
Artemisia biennis	5.6	0.2	1.1	16.0	74.4	0.7	20:0 = 1.5	20:1= 0.3	22:0=0.5	5.9	92.1	1.4	22=0.6
Coreopsis drummondii	9.6	0.3	1.2	15.5	68.9		14:0 = 0.1	20:0= 0.5	Polar=3.9	9.9	85.6	0.5	Polar=3.9
Coreopsis tinctoria	9.4	0.3	1.4	10.0	77.8	0.3	14:0 = 0.1	20:0= 0.3	22:0=0.4	10.2	89.2	0.6	14=0.1

[a] = 6,9,12 isomer
[e] = 1963 seed sample [f] = 1964 seed sample

esters were also separated on a 6 ft × 3/16 in column containing 14% butanediol succinate on acid washed Celite (60–80 mesh), also operated at 205C and 25 ml/min helium.

Column chromatography employing a silver nitrate-silicic acid column (4) was used to confirm the relative amounts of saturated and monounsaturated methyl esters present in *Hackelia americanum* seed oil.

For the chromatography of the polyyne from *Artemisia biennis*, Brockmann Activity No. 2 neutral alumina was used, the polyyne being eluted with n-hexane-benzene (2:1).

Results and Discussion

The average weight of seeds, oil content and protein content (% N × 6.25) are given in Table I. All data are reported on a moisture-free basis.

The composition and properties of the oils investigated are given in Table II and Table III.

In the case of the methyl esters of *Coreopsis drummondii* only a small peak with the ECL expected for methyl eicosanoate was detected on the polyester columns. On the silicone column, a much larger peak was observed at ECL 20 (4.4% of the total area of peaks). Such polar fatty acid methyl esters as those of 12-hydroxy-*cis*-9-octadecenoic, 9-hydroxy-*trans*, *trans*-10,12-octadecadienoic, and 9,10-epoxy-octadecanoic acids have ECL's in the neighborhood of 20 on nonpolar columns, but much longer retention times on polar polyester columns (14) making small amounts difficult to detect on the latter column. Thus, since both epoxide and conjugated diene were detected by spectroscopic tests, it was suspected that such polar fatty acid methyl esters might account for the greater area of the ECL 20 peak observed on the silicone column compared to the small peak assigned to methyl eicosanoate on the polyester column. The total fatty acid composition of the oil was adjusted to include the polar esters detected only on the silicone column. The GLC iodine value was calculated assuming 1.7% conjugated dienoic esters indicated by spectroscopic means.

Similar calculations were applied in the case of the *Mentzelia lindleyi* methyl esters, where an analogous situation occurred.

Artemisia biennis seed oil was saponified and the unsaponifiable material isolated by the usual procedure for determining per cent unsaponifiables (8). The infrared spectrum of the unsaponifiable material exhibited many bands of which two sharp bands at 2150 and 2250 cm^{-1}. were of special interest, suggesting the presence of acetylenic bonds. The ultraviolet spectrum (in methanol) had two broad maxima at 223 and 314 mμ, a lesser maximum at 254 mμ, shoulders at 237 and 268 mμ, and a minimum at 258 mμ, very similar to the published spectrum of polyyne I (6). Some purification of this material was achieved by partitioning the crude unsaponifiables between n-hexane and 85% aqueous methanol, and then chromatographing the aqueous methanol soluble fraction on alumina. The yellow oil so obtained had an infrared spectrum almost identical to a ca. 50:50 *cis, trans* mixture of polyyne I (6). The elemental analysis and molecular weight of this chromatographed oil indicated a closer fit to $C_{14}H_{14}O_2$ than to $C_{13}H_{12}O_2$(I). However, the NMR spectrum of the chromatographed material exhibited all the peaks shown in the NMR spectrum of I (*cis, trans* mixture) (6), with the relative peak areas expected for I. Additional peaks in the aliphatic methylene and aromatic proton regions suggested some contamination still present. Further work on the purification and reactions of the polyyne from *Artemisia biennis* is in progress.

It was also found that washing *Artemisia biennis* seed oil with 85% aqueous methanol removed the polyyne plus the free fatty acids from the triglycerides. The yield of chromatographed polyacetylenic material was 3.4% based on seed oil, or about 1% polyyne present in the seeds.

The chaff, consisting mostly of involucre bracts, obtained during the cleaning and separation of the achenes from *Artemisia biennis*, was also found to contain polyacetylenic compounds. Extraction of the chaff with hot n-hexane, followed by a purification scheme the same as that employed for the isolation of the polyyne from the seed oil, afforded about 1% of material with an infrared spectrum corresponding to a *cis, trans* mixture of polyyne I [Since this work was submitted for publication, further column chromatography of the polyyne mixture from the chaff, using the same procedure as Bohlmann and his associates (6), has resulted in the separation of both *cis* and *trans* polyyne I, with identical infrared spectra to those reported by Bohlmann (6).]

Some data has already been published by others for *Delphinium ajacis* (16) *Lappula echinata* (4), *Galeopsis tetrahit* (3), *Coreopsis tinctoria* (1) and *Coreopsis drummondii* (1). Earle (19) has also recently obtained data for *Chenopodium rubrum, Chenopodium hydridum, Atriplex hortensis, Gypsophila paniculata, Saponaria vaccaria, Moldavica parviflora,* and *Cam-*

panula rapunculoides. The present data are in general agreement with previous work with some variations in oil content observed. Earle and Jones (1) have reported 11.2% HBr absorbing acids in *Coreopsis drummondii*, whereas the present work found only 3.9% polar acids, but such variations in oxygenated fatty acid content has been reported previously for *Chrysanthemum coronarium* (17).

The compositions of the oils examined are in general quite similar to existing commercial oils, suggesting that any utilization of the species reported here will depend on more favorable crop characteristics which these may possess. *Artemisia biennis* appears to represent an easily accessible source of polyyne I.

Atriplex hortensis may represent a useful source of protein, since the yield of seed per plant appears to be high. However, Salgues (18) has reported that the seed of this species may contain toxic materials.

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A Comparison of Participating Solvents During Ozonization[1]

D. J. MOORE, E. H. PRYDE and J. C. COWAN,
Northern Regional Research Laboratory,[2] Peoria, Illinois

Abstract

Sixteen different participating solvents and certain combinations thereof were evaluated for their effects on the conversion of methyl oleate to carbonyl compounds by ozonolysis. Depending upon the alcohol or carboxylic acid used as a single solvent, chemical reduction with zinc and acetic acid gave yields of 70–100%; catalytic hydrogenation with 10% Pd/C, 62–84%. When equimolar mixtures of an acid and a primary, unhindered alcohol were used, catalytic hydrogenation gave yields of 94–98%. In preparative scale experiments, catalytic hydrogenation gave 98% yields of methyl azelaaldehydate in the representative solvent combinations of 2-methoxyethanol/acetic acid and 1-butanol/propionic acid. When anhydrous calcium sulfate was used as a drying agent for aldehyde/alcohol solutions significant acetal was formed in the absence of other catalysts.

Introduction

NUMEROUS COMPOUNDS with varying degrees of polarity and functionality have been used as solvents for the ozonization of unsaturated fatty materials (1–13). Best results have been achieved from solvents described as reactive and participating. Of this type, methanol (10,11) reportedly gives the best yields of carbonyl compounds; however, methanol has certain disadvantages as an ozonization medium for other than small-scale or exploratory investigations. It has high volatility and poor solubilizing properties; furthermore, the by-product methyl acetals and methyl esters complicate purification by distillation. Consequently, we sought a more suitable solvent system for preparative-scale ozonizations. We investigated various solvents that could be classified as reactive and participating. Criteria for such solvents were boiling points, solubilizing properties, and predicted reactivity. Four properties were measured for each solvent system: miscibility with methyl oleate, volatility, ozone absorption, and carbonyl yield.

Ozonization and Reduction

Ozonization procedures used have been previously reported (10). All alcohols were commercial grade, dried and distilled from potassium hydroxide. Acids were also commercial grade and used without further purification. The methyl oleate (Applied Science Laboratory) contained approximately 91% of its monounsaturation in the C_9 position as shown by oxidative cleavage (5).

Chemical Reduction

Ozonization in 2-Methoxyethanol. Methyl oleate (15.41 g 0.052 mole) and 2-methoxyethanol (242.0 g) were cooled to 10C in a reaction flask. Oxygen, containing 2–3% ozone, was bubbled through the mixture until a rapid increase in ozone concentration in the exit gases, as determined by a Welsbach Model C ozone meter, was noted. The ozone consumption was 108% of theory, and loss of volatile solvent was less than 0.5%/hr.

After 10 g of glacial acetic acid was added to the reaction mixture, 5 g of zinc dust was added slowly with stirring, and the reaction flask was immersed in a water bath to maintain the temperature at 20–25C. An aliquot of the reaction mixture then gave a negative peroxide test with potassium iodide in glacial acetic acid and was filtered through diatomaceous earth to remove excess zinc and zinc salts. Both reaction flask and filter cake were washed with 500 ml of methylene chloride, and the combined filtrate was washed with water until neutral to acid-alkali test paper. After drying, the solvent was removed on a rotary evaporator at <25C and reduced pressure. Carbonyl yield was 97% as determined by GLC procedure outlined below.

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[2] A laboratory of the No. Utiliz. Res. and Dev. Div., ARS, USDA.