102. *The Non-saponifiable Matter of Shea Nut Fat. Part IV.* A New Tetracyclic Diethenoid Alcohol, Butyrospermol.*

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Although it has been studied intensively for a number of years, the structure of β-amyrin, parent of a considerable number of naturally occurring members of the triterpene series, has yet to be completely elucidated. The determination of the structure of the tetracyclic alcohol, basseol, isolated from alleged shea nut fat in 1934 and subsequently shown to be cyclised easily to β-amyrin, is thus of considerable importance. In order to obtain fresh supplies of basseol, the

* Part II, Beynon, Heilbron, and Spring, *J.*, 1937, 987; Part III, *idem*, *Nature*, 1938, **142**, 434.

non-saponifiable portion of shea nut fat has been re-examined with the result that the acetate of an isomeric alcohol, butyrospermol, has been isolated from the fractions of the acetylated material which should have contained basseol acetate. Butyrospermol, like basseol, is a secondary alcohol containing two ethylenic linkages, one of which is extremely readily hydrogenated but, in sharp contrast, it cannot be converted into β-amyrin, and on ozonolysis it gives acetone as the volatile fragment instead of the formaldehyde obtained from basseol.

In Part I (Heilbron, Moffet, and Spring, *J.*, 1934, 1583), the isolation of a tetracyclic diethenoid alcohol, basseol, from the non-saponifiable matter of shea nut fat was described. Originally there was some doubt as to whether the new alcohol belonged to the steroid or the triterpene series but subsequently (Beynon, Heilbron, and Spring, *J.*, 1937, 989) its classification as a triterpene alcohol was proved conclusively by the discovery of its remarkable property of isomerising in the presence of acidic reagents to the well-known mono-unsaturated pentacyclic triterpene alcohol, β-amyrin. Investigation of the structure of basseol immediately assumed much importance since it seemed to afford the opportunity of obtaining information concerning the carbon skeleton of the β-amyrin ring system which has hitherto been extremely difficult to adduce. Moreover, since as a result of the brilliant researches of Ruzicka and his collaborators, the relationship between β-amyrin and a considerable number of triterpene alcohols and acids, now known as the β-amyrin or oleanolic acid group, has been clearly established (for a summary of the interconversions in this group, see Spring, *Ann. Reports*, 1941, **38**, 191), elucidation of the basseol structure, and hence that of β-amyrin, would furnish the key to the whole group.

Work was continued in various directions on the structure of basseol in these laboratories, but owing to its interruption by the war much remained incomplete. Selenium dehydrogenation of basseol was reported (Beynon, Heilbron, and Spring, *Nature*, 1938, **142**, 434) to give a phenanthrene homologue (probably a trimethylphenanthrene) which appeared to be identical with that obtained by Ruzicka, Hösli, and Ehmann (*Helv. Chim. Acta*, 1934, **17**, 442) by selenium dehydrogenation of hederagenin, a member of the β-amyrin group, and later identified (Ruzicka and Smith, *Chem. and Ind.*, 1938, 1210) as 1 : 2 : 6-trimethylphenanthrene. Later attempts to reinvestigate the dehydrogenation of basseol were less successful and it is not impossible that the material under investigation was in fact an impure sample.

In view of the potential importance of the problem this work was resumed as soon as possible after the cessation of hostilities, a lapse of six years. Two samples of shea nut fat were obtained, one (Sample A) from a commercial source with the assistance of the Ministry of Food, the other (Sample B) directly from West Africa through the Crown Agents for the Colonies.

Sample A was saponified in bulk with alcoholic potassium hydroxide giving about 6% of non-saponifiable matter. This was acetylated with boiling acetic anhydride, and a large crop of sticky, semi-crystalline material separated on cooling. The mother-liquors were cooled to below 0° whereupon a smaller crop of solid separated which in previous work had been found to consist essentially of basseol acetate (m. p. 141°, $[\alpha]_D^{18°}$ +22°).

This second crop was crystallised twice from ethyl acetate yielding long prisms, m. p. 139—141°, $[\alpha]_D^{17°}$ +23°, which gave a brown colour with strong green fluorescence in the Liebermann–Burchard test, thus corresponding in superficial properties to basseol acetate (see Table I). Observation of the melting point on the Kofler micro-melting point stage, however, revealed that although the bulk of the material melted at about 140°, crystalline fragments persisted in the melt above this temperature, finally disappearing at 180—200°. Repeated crystallisation eventually gave a least soluble fraction, m. p. 146·5—147·5°, $[\alpha]_D^{20°}$ +11° (±2°), giving the same colour reaction in the Liebermann–Burchard test as the impure material. Further crystallisation brought about no change in either melting point or specific rotation. Chromatography of the crude acetate ($[\alpha]_D$ +23°) gave a least strongly adsorbed fraction, $[\alpha]_D^{19°}$ +11°, later fractions having increasingly higher positive rotations and extended melting points similar to the starting material. The *acetate*, m. p. 146·5—147·5°, $[\alpha]_D^{20°}$ +11°, thus appeared to be a pure compound quite distinct from, but isomeric with, basseol acetate.

Hydrolysis of the acetate gave an *alcohol*, $C_{30}H_{50}O$, and thence a *benzoate*. Oxidation of the alcohol either under mild conditions with chromium trioxide or by the Oppenauer method using benzoquinone as the hydrogen acceptor (Wettstein, *Helv. Chim. Acta*, 1940, **23**, 388) gave a *ketone* (light absorption, $\lambda_{max.}$ 2650 A., ε = 500). Titration of the acetate with perbenzoic acid revealed the presence of two ethylenic linkages, and the absence of any high-intensity light absorption above 2200 A. indicated that these were not conjugated. When the alcohol was hydrogenated using a palladium on calcium carbonate catalyst at room temperature and pressure only one molecular proportion of hydrogen was absorbed to give the *dihydro-alcohol*, from which were obtained the *dihydro-acetate* and *-benzoate*. Oxidation of the dihydro-

alcohol with chromium trioxide gave a *dihydro-ketone* which, on reduction with sodium in alcohol, regenerated the dihydro-alcohol, identified as its acetate. The ease of hydrogenation of the alcohol under such mild conditions is noteworthy, the use of a platinum catalyst and acetic acid solutions having been found necessary by previous workers with similar compounds (*e.g.*, basseol, Beynon, Heilbron, and Spring, *loc. cit.*; euphol, Newbold and Spring, *J.*, 1944, 249; cryptosterol, Marker, Wittle, and Mixon, *J. Amer. Chem. Soc.*, 1937, **59**, 1368). The dihydro-acetate (also obtained by hydrogenation of the acetate in ethyl acetate solution at room temperature and pressure with a platinum catalyst) showed only one double bond on perbenzoic acid titration and gave a pale yellow colour with tetranitromethane. In the Liebermann–Burchard test it gave a pale yellow colour with green fluorescence.

As can be seen from Table I, the compounds now described differ noticeably in their physical properties from basseol and its derivatives. It was not possible to carry out mixed melting points between comparable compounds of the two series since all available samples of basseol and its esters were found to have changed on keeping.

The physical evidence given in Table I shows fairly conclusively that the new alcohol, tentatively named butyrospermol (from the name of the shea tree, *Butyrospermum parkii*, formerly *Bassia parkii*), differs from basseol. The chemical evidence removes any shadow of doubt as to this difference.

TABLE I.

	Basseol.		Butyrospermol.	
	M. p.	$[\alpha]_D$.	Micro-m. p.	$[\alpha]_D$ ($\pm 2°$).
Alcohol	109·5° [1]	−11·9°	111—113°	−12°
Acetate	141 [1]	+22·4	146·5—147·5	+11
Benzoate	130 [1]	—	130—133	+29·5
Ketone	73—74 [3]	[+19·7 [4]]	82·5—84	−40·5
Dihydro-alcohol	Oil [2]	—	114—116	−14
Dihydro-acetate	119—120 [2]	+32·5	137—139	+13·5
Dihydro-benzoate	156 [2]	+48·1	138—139	+30·5
Dihydro-ketone	Oil [3]	—	80·5—81	−43

[1] Heilbron, Moffet, and Spring, *loc. cit.*
[2] Beynon, Heilbron, and Spring, *loc. cit.*
[3] Prepared from the corresponding alcohol by distillation from copper bronze powder (unpublished work).
[4] Recently determined on a surviving sample.

As has already been mentioned, the most striking reaction of basseol was its ready isomerisation to β-amyrin in the presence of acids. This behaviour is in no way paralleled by the new alcohol, and in no case has β-amyrin been detected in any reaction product from pure butyrospermol. The acetate of the latter can be crystallised unchanged from boiling acetic acid, but it is almost insoluble in formic acid, with which it forms an oil on heating, resolidifying unchanged on cooling. Butyrospermyl acetate, on standing at room temperature in solution in chloroform saturated with dry hydrogen chloride, is not recovered unchanged, but gives rise to a compound containing chlorine with a similar melting point to the original acetate but a higher positive specific rotation. This product has not yet been fully investigated. Basseol acetate under these conditions gave β-amyrin in up to 90% yield (Beynon, Heilbron, and Spring, *loc. cit.*).

While only preliminary degradation experiments have been carried out on butyrospermol, these indicate a further profound difference in structure from basseol. Ozonolysis of butyrospermyl acetate has given a 64% yield of acetone, estimated as its 2 : 4-dinitrophenylhydrazone, in contrast to the 20% yield of formaldehyde obtained from basseol (Beynon, Heilbron, and Spring, *loc. cit.*). No volatile fragment was produced on ozonolysis of dihydrobutyrospermyl acetate.

Butyrospermol is, therefore, probably a tetracyclic diethenoid secondary alcohol. Of the two double bonds, one is present in an *iso*propylidene group, and is very easily hydrogenated, the other is not readily hydrogenated but it reacts with perbenzoic acid. The empirical formula, $C_{30}H_{50}O$, suggests that it may be classed as a triterpene alcohol and it shows many similarities to euphol (Newbold and Spring, *J.*, 1944, 249).

Application of the method of molecular rotation differences (Barton and Jones, *J.*, 1944, 659) to butyrospermol and euphol and to their dihydro-derivatives reveals some similarity between these and agnosterol. It must be observed that in the absence of the ketone values, all the above could be associated with the lupeol–betulin group, but such an association is excluded in the case of butyrospermol.

TABLE II.

Substance.	M_D. Alcohol.	Acetate. (1)	Benzoate. (2)	Ketone. (3)	Δ_1.	Δ_2.	Δ_3.
α- and β-Amyrin group [1]	—	—	—	—	+ 6°	+145°	+ 60°
Lupeol–betulin group [1]	—	—	—	—	+ 70	+200	+140
Basseol [2]	− 51°	+ 94°	—	[+ 85° [3]]	+145	—	+136 [3]
Butyrospermol	− 51	+ 51	+180°	−170	+102	+231	−119
Dihydrobutyrospermol	− 60	+ 56	+164	−182	+116	+224	−122
Euphol [4]	+132	+211	[+329 [5]]	—	+ 79	[+197]	—
Dihydroeuphol [4]	+116	+165	—	—	+ 49	—	—
Agnosterol [1]	+301	+424	+549	—	+123	+248	—
Dihydroagnosterol [1]	+260	+393	—	—	+133	—	—

[1] Barton and Jones, *loc. cit.* [2] Heilbron *et al.*, *loc. cit.* [3] See Table I, footnote 4.
[4] Jeger and Krusi, *Helv. Chim. Acta*, 1947, **30**, 2047. [5] Rotation determined in pyridine solution.

It can be suggested, however, that for these compounds neither the ring carrying the hydroxyl group nor the adjacent ring contains a double bond, since no anomalous effects due to any possible vicinal action are observable. A second point that emerges is the small and negative change in the molecular rotation that is observed on hydrogenation of the reducible double bond in butyrospermol, euphol, and agnosterol (Table III) as compared with the much larger differences in the lupeol–betulin group (Barton and Jones, *loc. cit.*).

TABLE III.

Substance.	M_D. Triterpene.	Dihydro-compound.	Δ.
Agnosterol	+301°	+260°	− 41°
,, acetate	+424	+393	− 31
Euphol	+132	+116	− 16
,, acetate	+211	+165	− 46
Butyrospermol	− 51	− 60	− 9
,, acetate	+ 51	+ 56	+ 5
,, benzoate	+180	+164	− 16
Basseol acetate	+ 94	+155	+ 61
Lupeol (mean value) [1]	—	—	−190

[1] Barton and Jones, *loc. cit.*

Sample B of the shea nut fat has as yet only been saponified in small batches. The total yield of non-saponifiable matter was of the order of 3% of which more than half was rubbery material, insoluble in boiling acetic anhydride. A small quantity of butyrospermyl acetate was obtained by crystallisation of the mixed acetates.

Two independent samples of shea nut fat have thus been found to contain the new alcohol, butyrospermol, in place of the basseol isolated hitherto. [Bauer and Moll (*Fette und Seifen*, 1939, **46**, 560) claim to have isolated basseol during an investigation of the non-saponifiable matter of shea nut fat, but since no detailed examination of its properties was made it is possible that this material was impure butyrospermol.] At present no explanation can be offered to account for the absence of basseol and the presence of butyrospermol, but it is hoped that investigation of a series of samples of shea nut fat obtained from kernels of varying states of maturity and from different geographical areas may bring to light some new facts, possibly the existence of more than one species at present classed under the common name of *Butyrospermum parkii*, as is maintained by some botanical authorities (Dalziel, " The Useful Plants of Tropical West Africa ", p. 350; A. Chevalier, Pierre MS.; Yates, *Herb. Kewensis*).

Investigations have been made (*Bull. Imp. Institute*, 1930, **28**, 130; 1931, **29**, 417; 1932, **30**, 282; 1933, **31**, 334; 1935, **33**, 271; 1936, **34**, 437) into the seasonal and geographical variations in shea nut fat, primarily with regard to its superficial properties, such as iodine value, saponification value, percentage of non-saponifiable matter, etc., in an attempt to correlate these variations with some botanical feature. No correlation has been possible as a result of this work, large variations in properties being found in fat obtained from kernels from the same tree in different seasons, or from kernels from different trees with the same superficial botanical features. Two facts, however, have emerged from these investigations. First, that trees from the eastern (Sudanese) end of the geographical range of the shea tree yield a fat with a lower non-saponifiable content than that from the trees from the western regions; secondly, that as the kernel ripens, the percentage of non-saponifiable matter decreases, but that the absolute quantity in a kernel remains constant during its development.

Experimental

(All rotations are in chloroform in a 1 dcm. tube. All melting points are on the Kofler block and corrected unless specifically stated.)

Isolation of Butyrospermol from the Non-saponifiable Matter of Shea Nut Fat.—(a) *From sample A.* The non-saponifiable matter (1·4 kg.) was heated under reflux for 4 hours with acetic anhydride (7 kg.). The hot solution was decanted from a small quantity of insoluble grey sludge and allowed to cool overnight to room temperature, when a massive crop of sticky solid separated. This was filtered off, pressed free of acetic anhydride, and washed well with water (wt. after drying in a vacuum, 540 g.). The filtrate was cooled to below 0° for two days; a further crop of sticky brown solid had then separated. This was filtered off, washed with a little cold ethanol, and dissolved in hot ethyl acetate (250 c.c.), and the solution cooled, whereupon a mass of white needles separated. After being filtered and washed with alcohol, the acetate (48 g.) had m. p. 136—139° (capillary, uncorr.), $[\alpha]_D^{18°}$ +22·5° (*c*, 1·95). In the Liebermann–Burchard test it gave a brown colour with strong green fluorescence. On the Kofler micro-melting point block the substance had m. p. 138·5—147·5°, with crystals persisting in the melt to 180° or higher. The acetate (10 g.), after five crystallisations from ethanol–ethyl acetate (10 : 3), gave pure *butyrospermyl acetate* (2·2 g.), m. p. 146·5—147·5°, $[\alpha]_D^{20°}$ +11° ±2° (*c*, 2·58). The compound gave a brown colour with tetranitromethane, while in the Liebermann–Burchard test it gave a pale brown colour with strong green fluorescence. For analysis a sample was sublimed in a high vacuum at 160° (Found: C, 81·75; H, 11·05. $C_{32}H_{52}O_2$ requires C, 82·0; H, 11·2%). Chromatography of the impure acetate, $[\alpha]_D^{18°}$ +22·5°, (2 g.) on a column of Peter Spence alumina (130 g.), followed by elution with benzene–light petroleum (b. p. 40—60°) (1 : 4), gave the following fractions (each 100 c.c.):

Fraction.	Wt. (mg.).	M. p.	$[\alpha]_D^{19°}$.	Fraction.	Wt. (mg.).	M. p.	$[\alpha]_D^{19°}$.
1	trace	—	—	7	110	137—190°	+22·5°
2	60	up to 140°	—	8	100	137—190	—
3	230	138—143	+11°	9	90	136—150	—
4	220	135—141	+16	10	80	137—150	—
5	160	135—141	+22	11	60	138—230	+26
6	130	134—141	—				

Fraction 3 proved to be essentially butyrospermyl acetate, while Fraction 4 after crystallisation also gave the pure acetate.

(b) *From sample B.* The non-saponifiable matter (19·2 g.) was heated under reflux with acetic anhydride (100 c.c.) for 5 hours. The hot solution was decanted from a sticky residue (10·8 g.), and cooled to below 0° for two days. The separated solid was filtered off, washed with a little methanol, and dissolved in hot ethyl acetate (60 c.c.) and ethanol (100 c.c.). On cooling to room temperature a crop of solid (3·5 g.), m. p. up to 185°, separated. This was rejected. Evaporation of the mother-liquors gave a second crop (1·05 g.), which on recrystallisation from ethyl acetate gave a small quantity of β-amyrenyl acetate, m. p. 234—239°, and a third crop (1·3 g.), which on crystallisation from ethyl acetate gave almost pure butyrospermyl acetate, m. p. 143—145°, $[\alpha]_D^{19°}$ +16°.

Butyrospermol and Butyrospermyl Benzoate.—Butyrospermyl acetate (0·4 g.) was hydrolysed by heating under reflux with 5% alcoholic potassium hydroxide (20 c.c.) for 2 hours, the reaction mixture was poured into water, and the product was isolated with ether. Crystallisation from aqueous methanol gave *butyrospermol* as fine needles, m. p. 111—113°, $[\alpha]_D^{19°}$ −12° (*c*, 1·77). For analysis a specimen was dried for 5 hours at 80°/0·5 mm. (Found: C, 84·35; H, 11·7. $C_{30}H_{50}O$ requires C, 84·45; H, 11·8%). Benzoylation in pyridine at room temperature gave *butyrospermyl benzoate*, which crystallised from benzene–ethanol as leaflets, m. p. 130—133°, $[\alpha]_D^{19°}$ +33·5° (*c*, 1·64). The analytical sample was dried for 5 hours at 80°/0·5 mm. (Found: C, 83·5; H, 10·0. $C_{37}H_{54}O_2$ requires C, 83·7; H, 10·25%).

Perbenzoic Acid Titration of Butyrospermyl Acetate.—The acetate (98·2 mg.) was kept at 0° in a chloroform solution of perbenzoic acid (25 c.c., 0·25N) for 3 days. Titration of an aliquot part with 0·1N-thiosulphate solution showed that 1·71 atoms of oxygen had been absorbed. After a further 2 days, 2·03 atoms and after a total of 12 days 2·11 atoms of oxygen had been absorbed.

Butyrospermone.—(a) *By chromium trioxide oxidation of butyrospermol.* The alcohol (0·3 g.) in benzene (10 c.c.) was shaken at room temperature for 2·5 hours with a solution of chromium trioxide (0·1 g.) in "AnalaR" acetic acid (5 c.c.) and water (5 c.c.). The product, isolated with ether, failed to solidify and was chromatographed on Peter Spence alumina (30 g.). Elution with benzene–light petroleum (b. p. 40—60°) (1 : 9) gave *butyrospermone* (267 mg.) crystallising from methanol–chloroform as small prisms, m. p. 82·5—84°, $[\alpha]_D^{19°}$ −40° (*c*, 1·51). An analytical sample was dried at 60°/1 mm. for 8 hours (Found: C, 84·9; H, 11·3. $C_{30}H_{48}O$ requires C, 84·8; H, 11·4%).

(b) *By Oppenauer oxidation of butyrospermol.* Butyrospermol (275 mg.), *p*-benzoquinone (800 mg.), and aluminium *tert.*-butoxide (300 mg.) were heated under reflux in dry benzene (25 c.c.) overnight. The reaction mixture was steam distilled until free from benzoquinone, and the residue extracted with ether. The ethereal layer was washed with dilute sodium hydroxide in brine and then with water. Evaporation gave an oily residue (153 mg.) which was chromatographed on a column of Peter Spence alumina (30 g.). Elution with benzene–light petroleum (b. p. 40—60°) (1 : 19) gave butyrospermone (123 mg.), crystallising from methanol–chloroform as prisms, m. p. 82—84°, $[\alpha]_D^{19°}$ −43·5° (*c*, 1·20), identical with that obtained in (a).

Dihydrobutyrospermol.—Butyrospermol (4 g.) in ethanol (50 c.c.) together with a palladium on calcium carbonate catalyst (0·5 g.; 10%) was shaken in hydrogen at atmospheric temperature and pressure. Hydrogen (231 c.c.) was rapidly taken up, absorption ceasing abruptly at this stage (theoretical volume for one double bond = 235 c.c.). After removal of the catalyst by filtration, the ethanol solution was evaporated to a small bulk; *dihydrobutyrospermol* (3·8 g.) then crystallised as fine needles, m. p. 114—116° (depressed to 105—109° on admixture with butyrospermol), $[\alpha]_D^{19°}$ −14°

(*c*, 1·66). A sample for analysis was crystallised from methanol, and dried at 80°/1 mm. for 5 hours (Found: C, 83·6; H, 11·9. $C_{30}H_{52}O$ requires C, 84·05; H, 12·25%). Acetylation with acetic anhydride in pyridine gave the *dihydro-acetate* crystallising from ethanol–ethyl acetate in thick needles, m. p. 136—139° (undepressed on admixture with butyrospermyl acetate), $[\alpha]_D^{19°}$ +14° (*c*, 2·0), also obtained by hydrogenation of butyrospermyl acetate in ethyl acetate solution in presence of platinic oxide. With tetranitromethane it gave a pale yellow colour, while in the Liebermann–Burchard test it gave a yellow colour with pale green fluorescence. A sample for analysis was crystallised from ethanol, and dried at 100°/1 mm. for 5 hours (Found: C, 81·65; H, 11·45. $C_{32}H_{54}O_2$ requires C, 81·65; H, 11·6%). *Dihydrobutyrospermyl benzoate*, prepared by benzoylation of the alcohol in pyridine, crystallised from ethanol in long laths, m. p. 138—139°, $[\alpha]_D^{19°}$ +30·5° (*c*, 1·65). A sample for analysis was dried at 100°/1 mm. for 5 hours (Found: C, 83·1; H, 10·7. $C_{37}H_{56}O_2$ requires C, 83·4; H, 10·6%).

Dihydrobutyrospermone.—Dihydrobutyrospermol (1·0 g.) in benzene (5 c.c.) and "AnalaR" acetic acid (30 c.c.) was treated with chromium trioxide (0·2 g.) in water (1 c.c.). After being left overnight at room temperature, the product was isolated with ether. *Dihydrobutyrospermone* (1 g.) crystallised from ethanol in needles, m. p. 80·5—81°, $[\alpha]_D^{19°}$ −43° (*c*, 1·30). A sample for analysis was dried at 60°/1 mm. for 4 hours (Found: C, 84·9; H, 11·85. $C_{30}H_{50}O$ requires C, 84·45; H, 11·8%).

Reduction of Dihydrobutyrospermone.—Dihydrobutyrospermone (100 mg.) in ethanol (25 c.c.) was treated with sodium (2 g.) and the reaction allowed to proceed vigorously under reflux, solution of the last traces of sodium being effected by warming. After being poured into much water the product was extracted with light petroleum (b. p. 40—60°). The residue obtained by evaporation of the solvent was acetylated at room temperature in pyridine solution, and the product isolated by evaporation of the pyridine in a vacuum. Two crystallisations from ethanol gave dihydrobutyrospermyl acetate (60 mg.), m. p. 136—138° (undepressed on admixture with that previously obtained), $[\alpha]_D^{21°}$ +10° (*c*, 1·34).

Ozonolysis of Butyrospermyl Acetate.—Butyrospermyl acetate (2·0 g.) in carbon tetrachloride (30 c.c.) was treated with ozonised oxygen at 0° for 45 minutes (equivalent to 3 mols. of ozone). The carbon tetrachloride solution was evaporated to dryness in a vacuum, the residue dissolved in "AnalaR" acetic acid (30 c.c.), diluted with water (100 c.c.), and steam distilled until 100 c.c. of distillate had been collected. The distillate was treated with an excess of aqueous 2 : 4-dinitrophenylhydrazine sulphate solution and the voluminous yellow precipitate of acetone 2 : 4-dinitrophenylhydrazone filtered off and dried in a vacuum (wt. 0·65 g.; 64% yield). After one crystallisation from aqueous ethanol it had m. p. 124° (capillary, uncorr.) undepressed on admixture with an authentic sample.

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