

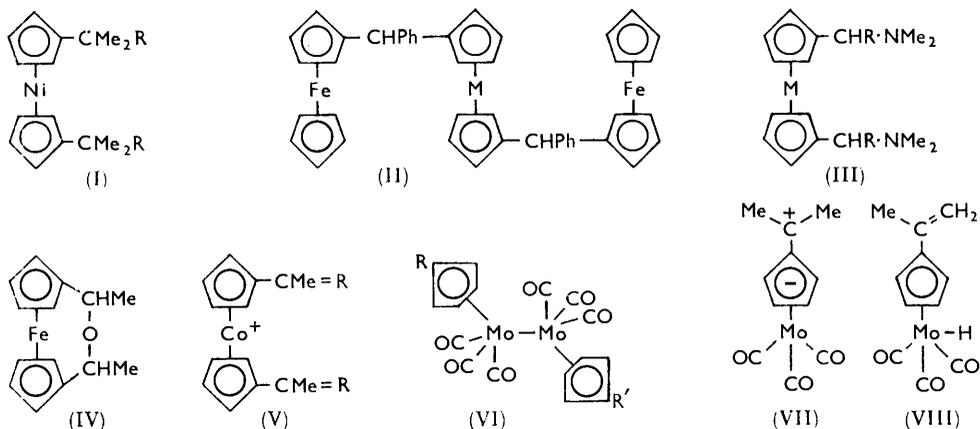
904. Some Substituted Cyclopentadienyl Derivatives of Nickel, Cobalt, Iron, Molybdenum, and Titanium.

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The applicability to other transition metals of methods developed for the synthesis of ferrocenes is demonstrated. The extension of the fulvene synthesis to aminofulvenes is described.

In preceding papers methods have been described for the synthesis of substituted ferrocenes directly from substituted cyclopentadienes, in particular from fulvenes,¹ diazo-cyclopentadiene,² and the anions³ of aminomethyl- and ethoxycarbonyl-cyclopentadienes. In the present paper we establish the applicability of these methods to those other transition metals which are known to afford stable cyclopentadienyl derivatives. This approach is particularly valuable for those compounds, *e.g.*, dicyclopentadienylcobalt(III) salts which cannot undergo electrophilic substitution. It should, however, be possible to modify substituents introduced by our direct method, and one example of such a reaction is included. Apart from the methylcyclopentadienyl and indenyl derivatives, the hydroxycyclopentadienides obtainable from cyclopentenones⁴ and cyclopentadienones⁵ are the only derivatives previously synthesised from these metals by such direct methods.

Fulvenes have previously been used by us in two ways: by addition to the :CR_2 group of alkyl or hydride ions to give alkylcyclopentadienide ions; and by proton abstraction to give alkenylcyclopentadienides. The former method has now been employed to obtain di(isopropylcyclopentadienyl)nickel (I; R = H) by reaction of dimethylfulvene and lithium aluminium hydride, and di-(α -dimethylbenzylcyclopentadienyl)nickel (I; R =



Ph) by reaction of dimethylfulvene and phenyl-lithium, followed in each case by treatment with tetrapyridinenickel chloride. We found the last reagent superior to nickel chloride or acetylacetonate for the preparation of dicyclopentadienylnickel. The product (I; R = Ph) was further converted by nitric oxide into nitrosyl- α -dimethylbenzylcyclopentadienylnickel. The method has also been applied to ferrocenylfulvene, which with

¹ Knox and Pauson, *J.*, 1961, 4610.

² Knox and Pauson, *J.*, 1961, 4615.

³ Osgerby and Pauson, *J.*, 1961, 4604.

⁴ Benson and Lindsay, *J. Amer. Chem. Soc.*, 1957, **79**, 5471.

⁵ Weiss and Hübel, *J. Inorg. Nuclear Chem.*, 1959, **11**, 42.

phenyl-lithium followed, respectively, by ferrous or cobaltous chloride afforded the trinuclear complexes (II; $M = \text{Fe}$ or Co), the cobalt complex being isolated, after oxidation by air as the corresponding cobalt(III) perchlorate, picrate, or reineckate.

A further extension of the fulvene method was found in the use of α -dimethylamino-fulvene, itself readily available by an improvement of Arnold and Zemlicka's method.⁶ With lithium aluminium hydride followed by ferrous or cobaltous chloride this fulvene gave the diamines (III; $R = \text{H}$, $M = \text{Fe}$ and Co), the latter being oxidised by air and isolated as the cobalticinium salt. The iron compound was shown to be identical with the known product.³ Its homologue (III; $R = \text{Me}$, $M = \text{Fe}$) was obtained similarly by employing methyl-lithium in place of the hydride. The dimethiodide of this homologue is readily hydrolysed to the bridged ether (IV; two stereoisomers). This and other reactions of the amines (III; $M = \text{Fe}$) will be described in detail later.

The isopropenylcyclopentadienyl anion, derivable from dimethylfulvene and sodamide, was likewise employed to obtain a cobalt derivative (V; $R = \text{CH}_2$). The value of this approach is illustrated by the further transformation of this product into di(acetylcyclopentadienyl)cobalt(III) salts on ozonolysis. The stability of the parent dicyclopentadienylcobalt(III) ion to ozone had previously been demonstrated.⁷

A further use of fulvenes appeared possible in their direct reaction with metal carbonyls. Molybdenum carbonyl was the most promising of these submitted to preliminary trial with dimethylfulvene. The products of the same reaction have recently been reported in some detail by Abel *et al.*⁸ and our own results are recorded only in so far as they differ from theirs. In 1,2-dimethoxyethane⁸ or similar solvents the sole product from dimethylfulvene and molybdenum carbonyl is the "saturated" binuclear product (VI; $R = R' = \text{Pr}^i$) whose formation must involve hydrogen abstraction from the solvent. Our initial experiments, however, were conducted in the absence of solvent (other than excess of fulvene) at 120°. The product obtained under these conditions gave eight bands on chromatography, all yielding crimson solids with very similar infrared spectra on elution. The first of these is due to the carbonyl (VI; $R = R' = \text{Pr}^i$), identical with that product obtained under the conditions of Abel *et al.*⁸ The second and largest single fraction was shown to be the unsymmetrical, monounsaturated product (VI; $R = \text{Pr}^i$, $R' = \text{CMe}:\text{CH}_2$) by analysis, molecular-weight determination, and quantitative hydrogenation to the saturated product (VI; $R = R' = \text{Pr}^i$). Although the other fractions have not been examined in detail, it appears reasonable to assume that the third is the diene (VI; $R = R' = \text{CMe}:\text{CH}_2$), and that the later fractions are polymers derived from this and the above unsymmetrical product. The formation of these unsaturated derivatives may be compared with the formation of similar products from fulvenes by our sodamide method.¹ Although the mechanism of the present reaction is unknown, the formation of these products may be understood by considering the hypothetical first intermediate (VII) which would be expected to isomerise to the hydride (VIII). Such hydrides are known to decompose⁹ below the reaction temperature used here, giving the dimers (VI). The hydridic hydrogen is not liberated¹⁰ as molecular hydrogen in the presence of olefins, and in the present case just sufficient hydrogen is available from this source to produce the unsymmetrical product (VI; $R = \text{Pr}^i$, $R' = \text{CMe}:\text{CH}_2$) or equimolecular amounts of the saturated and diunsaturated analogues.

In an extension of the fulvene method, diazocyclopentadiene was shown² to serve as a source of diarylazoferrocenes. This process has now been applied in preparations of 1,1'-diphenylazocobalticinium chloride and diphenylazocyclopentadienyltitanium dichloride.

⁶ Arnold and Zemlicka, *Coll. Czech. Chem. Comm.*, 1960, **25**, 1302.

⁷ Fischer, *Angew. Chem.*, 1955, **67**, 475.

⁸ Abel, Singh, and Wilkinson, *J.*, 1960, 1321.

⁹ Fischer, Hafner, and Stahl, *Z. anorg. Chem.*, 1955, **282**, 47.

¹⁰ Sternberg and Wender, *Chem. Soc. Special Publ.*, 1959, No. 13, p. 35.

EXPERIMENTAL

For general directions see the preceding papers.

Improved Preparation of Dicyclopentadienylnickel.—A slurry of tetrapyrindinenickel chloride (20 g., 0.045 mole) in tetrahydrofuran (40 ml.) was added to sodium cyclopentadienide [prepared from sodium wire (2.07 g., 0.09 g.-atom) and cyclopentadiene (6.6 g., 0.1 mole)] in tetrahydrofuran (100 ml.) under nitrogen. The mixture was stirred at room temperature for 30 min., then evaporated to dryness under reduced pressure. From the residue, the product (4.83 g., 57%) may be isolated by sublimation or by extraction with ligroin or ether, followed by rapid concentration and cooling to -70° ; dicyclopentadienylnickel crystallises as green needles, m. p. $172-173^\circ$ (decomp.) (Found: C, 63.5; H, 3.5. Calc. for $C_{10}H_{10}Ni$: C, 63.6; H, 5.3%). From larger runs yields up to 70% were obtained and isolation was conveniently effected by Soxhlet extraction.

1,1'-Di(isopropylcyclopentadienyl)nickel (I; R = H).—Dimethylfulvene (2.12 g., 0.02 mole) in ether (25 ml.) was added dropwise to a stirred suspension of lithium aluminium hydride (0.19 g., 0.02 mole) in ether (10 ml.) at room temperature, under nitrogen. A slurry of tetrapyrindinenickel chloride (4.46 g., 0.01 mole) in tetrahydrofuran (40 ml.) was then added and the mixture stirred for 1 hr. Evaporation *in vacuo* left a brown gum which was chromatographed in ligroin on alumina (35 g.). This solvent eluted *1,1'-di(isopropylcyclopentadienyl)nickel* as a green, air-sensitive liquid (0.99 g., 35%), which was further purified by distillation at 128° (bath)/0.3 mm. (Found: C, 70.1; H, 8.2. $C_{16}H_{22}Ni$ requires C, 70.4; H, 8.1%).

1,1'-Bis-(α -dimethylbenzylcyclopentadienyl)nickel (I; R = Ph).—Dimethylfulvene (2.12 g., 0.02 mole) in ether (10 ml.) was added dropwise with stirring and occasional cooling to phenyllithium [prepared from bromobenzene (3.5 g., 0.021 mole) and lithium (0.33 g., 0.05 g.-atom)] in ether under nitrogen. Tetrapyrindinenickel chloride (4.46 g., 0.01 mole) in tetrahydrofuran (50 ml.) was then added and the mixture stirred at room temperature for 1 hr. Evaporation of the filtered mixture *in vacuo* gave a gum (2.87 g.) which was dissolved in ligroin. Concentration and cooling of the ligroin to -70° yielded green needles of *1,1'-di-(α -dimethylbenzylcyclopentadienyl)nickel* (2.68 g., 63%), m. p. $109-110^\circ$, soluble in the common organic solvents (Found: C, 78.9; H, 7.3. $C_{28}H_{30}Ni$ requires C, 79.1; H, 7.1%).

Nitrosyl-(α -dimethylbenzylcyclopentadienyl)nickel.—A ligroin solution (75 ml.) of the preceding product (2.12 g.) was saturated with nitric oxide, then filtered and evaporated *in vacuo*. The residue (0.81 g.) was chromatographed on alumina with ligroin as solvent. Distillation of the main band afforded *nitrosyl-(α -dimethylbenzylcyclopentadienyl)nickel* (0.69 g., 51%) as a dark red liquid, b. p. $146^\circ/0.3$ mm. (Found: C, 61.7; H, 5.4; N, 5.0. $C_{14}H_{15}NNiO$ requires C, 61.8; H, 5.6; N, 5.2%).

1,1'-Di-(α -ferrocenylbenzyl)ferrocene (II; M = Fe).—Freshly prepared α -ferrocenylfulvene³ (1.14 g.) in ether (25 ml.) was added dropwise to stirred phenyllithium [prepared in ether from bromobenzene (1.60 g., 0.01 mole) and lithium (0.2 g., 0.029 g.-atom)] under nitrogen. A pale yellow precipitate was formed almost instantly. After 1.5 hours' stirring, ferrous chloride [from ferric chloride (0.49 g., 0.003 mole)] in tetrahydrofuran (20 ml.) was added, and stirring continued for 66 hr. The mixture was poured into ice-water (200 ml.) and extracted with ether (3 \times 50 ml.), and the combined extracts were washed with water, dried (Na_2SO_4), and evaporated under reduced pressure. The oily residue was chromatographed in ligroin-benzene (4 : 1) on alumina. Ligroin eluted starting material (0.061 g., 5.5%), and ligroin-benzene (4 : 1) then eluted *1,1'-di-(α -ferrocenylbenzyl)ferrocene (II; M = Fe)* (0.32 g., 21%) which crystallised from ligroin at 0° as a fine powdery yellow solid, m. p. $194-196^\circ$ (Found: C, 72.5, 72.3, 72.2; H, 5.3, 5.7, 5.7. $C_{44}H_{38}Fe_3$ requires C, 72.0; H, 5.2%).

1,1'-Di-(α -ferrocenylbenzyl)cobalticenium Salts (II; M = Co^+).—The above experiment was repeated with anhydrous cobaltous chloride (0.65 g., 0.005 mole) in place of the ferrous salt. The reaction mixture was poured into ice and hydrochloric acid with free access of air. The resultant aqueous solution containing the *1,1'-di-(α -ferrocenylbenzyl)cobalticenium chloride* was separated from the organic phase. The corresponding picrate, reineckate, and perchlorate were readily precipitated from this solution, but attempts to recrystallise these salts led to partial decomposition.

α -Dimethylaminofulvene.—Tetramethylformamidinium perchlorate¹¹ (10 g., 0.05 mole) was added in small portions to cyclopentadienylsodium [from ultrasonically dispersed sodium

¹¹ Arnold, *Coll. Czech. Chem. Comm.*, 1959, **24**, 760.

(1.4 g., 0.06 g.-atom) and an excess of cyclopentadiene] in tetrahydrofuran (180 ml.). After 12 hours' stirring, methanol (20 ml.) was added and the mixture poured into water (250 ml.) and extracted with ether. The dried (Na_2SO_4) extracts were evaporated under reduced pressure, leaving the crude fulvene (4.7 g., 78%) as a flaky yellow solid, m. p. 61—64°. Recrystallisation from ligroin raised this to m. p. 64—66° (lit.,⁶ m. p. 50—64.5°) (Found: C, 79.2; H, 9.1; N, 11.7. Calc. for $\text{C}_8\text{H}_{11}\text{N}$: C, 79.3; H, 9.2; N, 11.6%).

1,1'-Bis(1-dimethylaminoethyl)ferrocene (III; M = Fe, R = Me).—A solution of α -dimethylaminofulvene (1.6 g., 0.013 mole) in ether (20 ml.) was added dropwise to methyl-lithium [from lithium (0.28 g., 0.041 g.-atom) and methyl iodide (2.84 g., 0.02 mole)] in ether (40 ml.) under nitrogen. After 4 hours' stirring, anhydrous ferrous chloride [from ferric chloride (2.3 g., 0.014 mole)] in tetrahydrofuran (10 ml.) was added; the mixture began to reflux spontaneously and was stirred overnight. Methanol (10 ml.) was added and the whole then poured into water (200 ml.) and extracted with ether. The dried (Na_2SO_4) extracts were evaporated under reduced pressure and the viscous *product* (III; M = Fe, R = Me) (1.54 g., 71%) distilled at 120° (bath)/1 mm. (Found: C, 64.9; H, 8.3; N, 8.3. $\text{C}_{18}\text{H}_{28}\text{FeN}_2$ requires C, 65.8; H, 8.6; N, 8.5%). It forms an unstable dimethiodide.

1,1'-Bis(dimethylaminomethyl)ferrocene (III; M = Fe, R = H).—A solution of α -dimethylaminofulvene (1.21 g., 0.01 mole) in ether (10 ml.) was added dropwise to a stirred suspension of lithium aluminium hydride (0.4 g., 0.01 mole) in ether (20 ml.) under nitrogen at room temperature. The mixture was stirred for 2 hr., then ferrous chloride [from ferric chloride (1.29 g., 0.008 mole)] in tetrahydrofuran (20 ml.) was added and stirring continued overnight. Ice was added, the ether layer was separated, the aqueous layer extracted with ether, and the combined ether solutions were dried (Na_2SO_4) and concentrated *in vacuo*. The residual amber oil (0.86 g., 58%) consisted of 1,1'-bis(dimethylaminomethyl)ferrocene (III; M = Fe, R = H), identical with the product previously described³ as shown by its infrared spectrum and by the m. p.s and mixed m. p.s of the picrates and methiodides. It was further characterised by conversion of the dimethiodide with picric acid into the *dimethopicrate* which forms yellow needles from aqueous methanol, m. p. 228° (decomp.) (Found: C, 45.4; H, 4.4; N, 14.4. $\text{C}_{30}\text{H}_{34}\text{FeN}_8\text{O}_{14}$ requires C, 45.8; H, 4.4; N, 14.3%).

1,1'-Bis(dimethylaminomethyl)cobalticenium Salts (III; M = Co^+ , R = H).—The preceding experiment was repeated with cobaltous chloride (1 g., 0.008 mole) in place of ferrous chloride. After addition of ice, the organic phase was extracted exhaustively with dilute hydrochloric acid in presence of air, and the combined aqueous solutions were brought to pH 4. The aqueous solution was divided into two portions, from which the *reineckate* and *tetraphenylborate* were severally precipitated (total yield 78%). The *reineckate* formed a red powder from methanol. The *tetraphenylborate* crystallised as a pale yellow powder from methanol (Found: C, 81.7; H, 7.2. $\text{C}_{64}\text{H}_{68}\text{B}_2\text{CoN}_2$ requires C, 81.5; H, 6.95%).

1,1'-Di-isopropenylcobalticenium Salts (V; R = CH_2).—Freshly distilled dimethylfulvene (21 g., 0.2 mole) was added to sodamide (from 4.6 g. of sodium) in liquid ammonia (200 ml.) under nitrogen. The mixture was stirred for 0.5 hr., and tetrahydrofuran (200 ml.) was then added as the ammonia evaporated. After brief warming to remove most of the residual ammonia, cobaltous chloride (13 g.) was added and stirring continued overnight. The product was poured on ice and hydrochloric acid and extracted with ether until the extracts became colourless. The yellowish-brown aqueous solution was then divided into several portions. From these, the 1,1'-di(isopropenylcyclopentadienyl)cobalt(III) compound was precipitated with picric acid as the *picrate*, crystallising from water as orange needles, m. p. 122.5—123° (Found: C, 53.4; H, 4.4. $\text{C}_{22}\text{H}_{20}\text{CoN}_3\text{O}_7$ requires C, 53.1; H, 4.1%), and with ammonium perchlorate as the *perchlorate*. The latter forms as an orange microcrystalline precipitate, sparingly soluble in water from which it may be recrystallised with addition of a little saturated aqueous ammonium perchlorate. A sample, further purified by slow precipitation from its acetone solution with ether, did not melt sharply, but gradually liquefied above 145° (Found: C, 52.5; H, 5.62. $\text{C}_{10}\text{H}_{13}\text{ClCoO}_4$ requires C, 52.1; H, 4.9%). The compound was also precipitated as *tetraphenylborate* (yellow powder, crystallisable by slow precipitation from acetone solution with water), *reineckate* (tan, acetone-soluble powder), *tribromide* (orange gum), and a tri(or poly?)iodide (dark brown, microcrystalline).

1,1'-Diacetylcobalticenium Salts (V; R = O).—A stream of ozone (0.005 g. per min.) was passed for 1 hr. through a solution of 1,1'-di-isopropenylcobalticenium perchlorate (1 g., 0.0027 mole) in chloroform (25 ml.) at 0°. Water (50 ml.) was then added and the mixture

heated on a steam bath for 1 hr. The mixture was distilled until all the chloroform was removed. From an aqueous extract of the chloroform distillate, formaldehyde (54 mg.) was precipitated as its dimedone derivative, m. p. and mixed m. p. 185—186°. The cooled aqueous distillation residue was divided into two equal portions. Addition of a saturated solution of Reinecke's salt to one of these precipitated 1,1'-diacetylcobalticenium reineckate (0.52 g., 65%) crystallising from acetone as orange plates, m. p. 176—179° (with darkening from 155°) (Found: C, 36.4; H, 3.5; N, 14.0. $C_{18}H_{20}CoCrN_6O_2S_4$ requires C, 36.6; H, 3.4; N, 14.2%). The second portion gave a precipitate of the tetraphenylborate, which formed brown crystals (from acetone), m. p. 195—198° (decomp. from 165°) (Found: C, 77.2; H, 5.9. $C_{38}H_{34}BCoO_2$ requires C, 77.0; H, 5.8%). Both salts showed strong absorption at 1682 cm^{-1} (C=O) (KCl disc).

1,1'-Diphenylazocobalticenium Chloride.—Diazocyclopentadiene² (2.76 g., 0.03 mole) in ether (20 ml.) was added dropwise to a stirred solution of phenyl-lithium (0.03 mole) in ether (50 ml.) under nitrogen at -30°. A scarlet precipitate was formed. After 30 min. cobaltous chloride (1.94 g., 0.015 mole) was added and the mixture then stirred overnight, being allowed to warm gradually to room temperature. The solvent was removed *in vacuo*, 2*N*-hydrochloric acid (50 ml.) added, and the mixture heated to 100° for 10 min. The deep red solution was filtered hot and, on cooling, deposited 1,1'-diphenylazocobalticenium chloride (71 mg.). Recrystallisation from 2*N*-hydrochloric acid gave dark red, slightly hygroscopic crystals, m. p. 138—139° (decomp.) (Found: C, 60.9; H, 4.1; N, 12.5; Cl, 7.9. $C_{22}H_{18}ClCoN_4$ requires C, 61.0; H, 4.2; N, 12.9; Cl, 8.2%). From half of the filtrate the corresponding reineckate (1.63 g.) was precipitated. Recrystallisation from acetone gave orange-brown crystals, m. p. 217—219° (decomp. from 190°) (Found: C, 43.2; H, 3.2; N, 19.3. $C_{28}H_{24}CoCrN_{10}S_4$ requires C, 43.6; H, 3.4; N, 19.6%). The remainder was precipitated as the tetraphenylborate (1.45 g.) which formed brown plates (from acetone), decomp. >160° (Found: C, 76.8; H, 5.1; N, 7.6. $C_{46}H_{34}BCoN_4$ requires C, 77.1; H, 5.4; N, 7.8%). The total yield of 1,1'-diphenylazocobalticenium salts was 30%.

Di(phenylazocyclopentadienyl)titanium Dichloride.—To phenylazocyclopentadienyl-lithium (0.03 mole), prepared as above, a solution of titanium tetrachloride (2.84 g., 0.015 mole) in tetrahydrofuran (20 ml.) was added and the mixture stirred at room temperature overnight. The solvent was removed *in vacuo*, the residue triturated with benzene, and the benzene extract evaporated again, to leave a red gum (2.8 g., 42.5%) which crystallised with some difficulty from benzene-ligroin. Di(phenylazocyclopentadienyl)titanium dichloride was obtained as a brown solid, m. p. 198—204° (decomp. from 175°) (Found: C, 57.2; H, 3.8; N, 11.9. $C_{22}H_{18}Cl_2N_4Ti$ requires C, 57.8; H, 4.0; N, 12.3%).

Hexacarbonyl(isopropenylcyclopentadienyl)(isopropylcyclopentadienyl)dimolybdenum (VI; R = Prⁱ, R' = CMe.CH₂).—Dimethylfulvene (17 g., 0.16 mole) and dimolybdenum hexacarbonyl (17 g., 0.07 mole) were heated under nitrogen at 120° for 8.5 hr. Starting materials were removed *in vacuo*, and the residue was chromatographed in light petroleum (b. p. 40—60°) on alumina, with the same solvent as eluent. The first band afforded hexacarbonyldi(isopropylcyclopentadienyl)dimolybdenum (VI; R = R' = Prⁱ) as purple-red crystals, m. p. 155—156° (lit.,⁸ m. p. 163°), undepressed and identical in infrared spectrum with a sample prepared by the recorded method⁸ [Found: C, 45.9; H, 3.95%; *M* (cryoscopic in benzene *), 520. Calc. for $C_{22}H_{22}Mo_2O_6$: C, 46.0; H, 3.9%; *M*, 574]. Elution of the second band afforded hexacarbonyl(isopropenylcyclopentadienyl)(isopropylcyclopentadienyl)dimolybdenum as purple crystals (from light petroleum), m. p. 119° [Found: C, 46.2; H, 3.6%; *M* (cf. above), 500. $C_{22}H_{20}Mo_2O_6$ requires C, 46.2; H, 3.5%; *M*, 572]. Six further bands separated on the column and were eluted with ligroin, benzene, and finally ether. All showed strong peaks in the metal-carbonyl region near 1957 and 1905 cm^{-1} . They did not crystallise readily and the later fractions were undoubtedly polymeric. Three samples of the product (VI; R = Prⁱ, R' = CMe.CH₂) were hydrogenated at room temperature over 10% palladium-charcoal. In each case hydrogen uptake ceased after absorption of 1 molecule ($\pm 10\%$) [calculated after correction for a control value obtained similarly with the "saturated" product (VI; R = R' = Prⁱ)]. The hydrogenation product was identified by m. p., mixed m. p. and infrared comparison with the authentic product (VI; R = R' = Prⁱ).

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