of pseudohalides in tetrahydrofuran. The products were isolated by removing the solvent under reduced pressure and subsequent crystallisation from petroleum ether.

\[
\text{(C}_5\text{H}_5\text{)_2 MoOCl}_2 + 2kR \rightarrow \text{(C}_5\text{H}_5\text{)_2 MoOR}_2 + 2k\text{Cl} \]

where R may be CN, NCO, NCS and N\(_3\) and k is either potassium or sodium.

The compounds prepared vary from red brown to dark brown in colour and are stable in dry atmosphere. These compounds are non-volatile, soluble in common organic solvents and are hydrolysed by water, dilute acids and alkalies.

**Experimental**—All the reactions were carried out in dry, inert atmosphere. The solvents were dried and purified before use by conventional methods. Tetrahydrofuran was refluxed over potassium hydroxide followed by distillation in presence of lithium aluminium hydride.

To bicsyclopentadienyl molybdenum (VI) oxydichloride 1.1 g (0.0035 mole) in tetrahydrofuran (100 ml) was added anhydrous potassium thiocyanate 0.058 g (0.0052 mole). The mixture was refluxed at 70-80°C for 2 hours, cooled and filtered. The filtrate on evaporation under reduced pressure gave red brown residue which on repeated crystallisation from petroleum ether (60-80°C) gave red brown crystals of (C\(_5\)H\(_5\))\(_2\)MoO(NCS)\(_2\) yield—80%. Anal. calcd. for (C\(_5\)H\(_5\))\(_2\)MoO(NCS)\(_2\) : C, 40.22; H, 2.79; Mo, 26.81%. Found: C, 40.12; H, 2.61; Mo, 26.60%.

Molybdenum was estimated as oxinate and C, H were estimated by microanalytical methods. The i.r. spectra of the compounds recorded on Perkin-Elmer Model-137 Spectrophotometer in KBr medium showed the following absorption peaks:

\[
3010 \text{ cm}^{-1} \quad 1710 \text{ cm}^{-1} \quad 1540 \text{ cm}^{-1} \quad 1470 \text{ cm}^{-1} \quad 1160 \text{ cm}^{-1} \quad 1050 \text{ cm}^{-1} \quad 955 \text{ cm}^{-1} \quad 850 \text{ cm}^{-1} \]

where vs = very strong; s = strong; m = medium; w = weak.

The i.r. spectrum of bicsyclopentadienyl molybdenum (VI) oxydithiocyanate shows the usual peaks of C\(_5\)H\(_5\) group, viz., the frequencies at 3010 cm\(^{-1}\) (C–H stretching), at 1470 cm\(^{-1}\) (C–C stretching), at 1160, 1050 cm\(^{-1}\) (C–H in plane bending) and at 850 cm\(^{-1}\) (C–H out of plane bending). The peak at 960–45 cm\(^{-1}\) is due to metal-oxide, i.e., M=O linkage.

Metal thiocyanates (M–SCN) are indicated by C–N stretching vibration appearing at \(\geq 2100 \text{ cm}^{-1}\) and C–S stretching vibrations at 690–720 cm\(^{-1}\). The metal isothiocyanates (M–NCS) are indicated by C–N stretching at \(\leq 2100\) and C–S stretching at higher frequency \(780–860 \text{ cm}^{-1}\). In cyclopenta dienyl compounds, the later band \(780–860 \text{ cm}^{-1}\) may be masked by the strong absorption around \(850 \text{ cm}^{-1}\) due to C–H out of plane bending vibrations of the ring. However, the absence of any bands in the region 650–750 cm\(^{-1}\) and the presence of strong bands at \(850 \text{ cm}^{-1}\) and 2050 cm\(^{-1}\) strongly indicates the presence of M–NCS bonding structure. This is in accord with the observations of Burmeister\(^2\) that N coordination usually results in an increase in C–S and C–N stretching values relative to free ion values (\(\approx 749 \text{ cm}^{-1}\) and 2060 cm\(^{-1}\)).

Similar observations were made in i.r. spectra of the metal cyanate showing an intense band at \(1320 \text{ cm}^{-1}\) and this is consistent with the isocyanate structure\(^6\); i.e., M–NCO. Other pseudohalide complexes were prepared similarly and their analytical and i.r. data was in agreement with the calculated values.

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**CHEMICAL INVESTIGATION OF THE ESSENTIAL OIL OF PSEUDOSORGHUM GRASS**

The essential oil (34 g) was separated into acidic and neutral part by treating with an aq. sodium hydroxide solution (5%). Acidic portion was found to contain two components, one giving positive ferric chloride colour test while the other giving effervescence with sodium bicarbonate solution. Therefore, acidic portion was taken in ether and washed with saturated solution of sodium bicarbonate. The sodium bicarbonate insoluble part yielded the solid phenol, i.e., xanthovenylene\(^2\) (1·6 g) which was further confirmed by the preparation of its acetyl derivative and the soluble part yielded an
acid, i.e., 3-hydroxy-\(p\)-toluic acid\(^6\)\(^7\) (0.47 g); this too was further confirmed by the preparation of its acetyl derivative.

The neutral part (32 g) was chromatographed over neutral alumina grade II and three fractions were collected by eluting the column with petroleum ether, benzene and ether.

**Petroleum ether fraction**: (TLC five spots) of alumina on extensive chromatography over silicagel and silicagel impregnated with silver nitrate (15\%) afforded \(n\)-tridecane\(^8\) (3.5 g), decanone-4\(^9\)\(^10\) (0.86 g) and octanone-3 (0.53 g)\(^11\). All the three compounds have been identified on the basis of their physical, chemical and spectral data.

**Benzene fraction** (TLC, three spots): On extensive chromatography over active silicagel afforded two components, an aromatic ether (under investigation) and an ester, \(p\)-methoxy methyl cinnamate\(^12\)\(^13\), m.p. 87-88\(^\circ\).

**Ether fraction** (TLC, three spots): On extensive chromatography over active silicagel, afforded an open chain alcohol decanol-4. The structure has been confirmed on the basis of its physical, chemical and spectral data.

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**CHEMICAL EXAMINATION OF THE FLOWERS OF PEDALIUM MUREX**

*Pedalium murex* (Pedaliaceae) is a small flowering plant growing abundantly in the coastal regions of South India. Though there are reports\(^1\) about its therapeutic uses, no detailed examination of the plant as a drug has been done so far. The leaves of this plant have been shown to contain flavonoid compounds\(^2\). We have now examined the polyphenolic components of the flowers of this plant and report the results in this communication.

The fresh flowers, collected around Madurai University area, were repeatedly extracted with 95\% ethanol, till the extract was colourless. The combined extract after concentration was fractionated using neutral and basic lead acetate. The lead salt from the neutral lead acetate fraction after decomposition with hydrogen sulphide yielded a crude mixture of flavonoid compounds.

**Aglycones**: The residue from the decomposition of the neutral lead salt fraction was macerated repeatedly with ether to remove free aglycones and the ether extract after evaporation yielded a mixture of aglycones. Paper chromatography of the aglycone mixture using the B.A.W. as the solvent system (4:1:5, v/v/v) showed the presence of two flavonoids having \(R_f\) values 0.95 and 0.64. These were separated by preparative paper chromatography. The aglycone having the higher \(R_f\) value was found to be dinatin (5,7,4'-trihydroxy-6-methoxyflavone) by a detailed study of its U.V. spectrum and comparison with an authentic sample. The compound with lower \(R_f\) value was also studied in the same way and identified as quercetin (3,5,7,3',4'-pentahydroxyflavone).

**Glycosides**: The glycoside mixture obtained as the other insoluble fraction was found by paper chromatography (B.A.W., 4:1:5, v/v/v) to be a mixture of two compounds (\(R_f\) values 0.32 and 0.28). These were separated by preparative paper chromatography using the same solvent system. The glycoside (\(R_f\) value 0.32) on hydrolysis with 7\% sulphuric acid yielded glucose and quercetin (identified by paper chromatography). A detailed study of the U.V. spectra of the glycoside and the aglycone with and without the addition of the various shift reagents suggested its identity as quercetin-7-glucoside. Rigorous comparison of the natural material with authentic quercetin confirmed the identity. The slow-moving component of the glycoside mixture (\(R_f\) value 0.28) on hydrolysis with 7\% sulphuric acid yielded rhamnose in addition to quercetin and glucose. By a careful study of the U.V. spectra of the glycoside and aglycone with and without the addition of various reagents such as sodium acetate, sodium ethoxide,