

synthesis of the phenanthrenequinone trianion radical and comparison of its spectral properties with those of the trianion radical obtained from benzil. Thus, both routes produce a trianion radical which gives a five-line e.p.r. spectrum (coupling with the four starred protons) and absorbs in the visible region at 555 and 410 m μ .

The HMO calculations indicate that, whereas the NBMO being filled at the B₂⁻ and B₂⁼ stages is predominantly an α -diketone MO, that which accommodates the third electron is largely of the *cis*-stilbene type, with high spin density on the phenyl rings at the *ortho* and *para* positions. The similar fate of B₂⁼ and photoexcited *cis*-stilbenes⁴ suggests a possible parallel between reactions of highly electronated and photoexcited molecules.

Calculations for the oxalate system also show an MO at $\alpha - 0.73\beta$ which is vacant at the dianion stage. Again, excess potassium is found to convert the dianion, sodium oxalate, into a stable, purple trianion radical showing an e.p.r. singlet.



A detailed report of the e.p.r. spectra of these and other trianion radicals will be forthcoming at a later date.

(4) R. Srinivasan and J. C. Powers, *J. Am. Chem. Soc.*, **85**, 1355 (1963); F. B. Mallony, *et al.*, *ibid.*, **84**, 4361 (1962).

DEPARTMENT OF CHEMISTRY
THE UNIVERSITY OF TEXAS
AUSTIN, TEXAS 78712

NATHAN L. BAULD

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A Krypton Difluoride-Antimony Pentafluoride Complex¹

Sir:

Krypton difluoride has recently been prepared in these laboratories by an electric discharge method.² This compound, though manifestly less stable than xenon difluoride,³ might be expected to show an analogous chemistry. Recently it has been shown that xenon difluoride interacts with antimony pentafluoride to form a stable complex of composition XeF₂·2SbF₅,⁴ and now we report the preparation of the analogous complex KrF₂·2SbF₅. The compound is formed by treating krypton difluoride with antimony pentafluoride in glass or Kel-F. Reaction is complete at -20°, and if excess antimony pentafluoride is present, most of the white crystalline material formed dissolves to give a clear yellowish solution at room temperature. The white complex can be recovered from the solution by pumping away the solvent at 25°. Two separate specimens gave the following analytical results: (1) sample weight 0.875 g.; Kr, 15.7%; Sb, 42.4%; and F, 41.9%; (2) sample weight 0.189 g.; Kr, 14.2%; Sb, 44.2%; and F, 41.6%. KrF₂·2SbF₅ requires Kr, 15.1%; Sb, 43.8%; F, 41.0%.

(1) Based on work performed under the auspices of the U. S. Atomic Energy Commission.

(2) F. Schreiner, J. G. Malm, and J. C. Hindman, to be published.

(3) D. R. MacKenzie, *Science*, **141**, 1171 (1964).

(4) A. J. Edwards, J. H. Holloway, and R. D. Peacock, *Proc. Chem. Soc.*, 275 (1965).

The complex KrF₂·2SbF₅ is more stable and much less volatile than the parent difluoride. For example, a sample weighing 0.848 g. lost only 0.020 g. on pumping for 20 hr. at 25°. It melts at about 50°, but the liquid decomposes quickly to antimony pentafluoride, krypton, and fluorine. The complex also decomposes slowly in antimony pentafluoride solution at 25°. As expected, it is an extremely reactive material and ignites organic matter explosively. Solutions of KrF₂·2SbF₅ in antimony pentafluoride appear to attack Kel-F. Aqueous hydrolysis in basic or slightly acidic solution yields not only the expected krypton and oxygen, but also appreciable quantities of fluorine monoxide. The infrared spectrum of the complex shows a strong characteristic absorption band at 813 cm.⁻¹ and other strong, poorly resolved absorptions in the 600-700 cm.⁻¹ region.

Preliminary experiments show that krypton difluoride combines with arsenic pentafluoride at -78°, but the adduct is very unstable and we have been unable to isolate it.

CHEMISTRY DIVISION

ARGONNE NATIONAL LABORATORY
ARGONNE, ILLINOIS

HENRY SELIG
RAYMOND D. PEACOCK

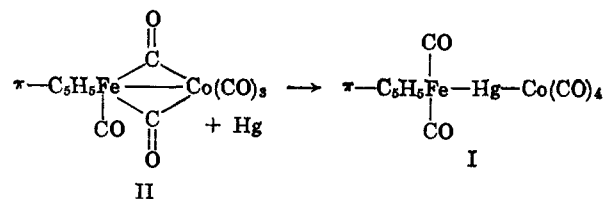
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An Organometal Carbonyl Containing Covalent Bonds from Iron to Mercury to Cobalt

Sir:

Mercury salts of binuclear metal carbonyls, such as Hg[Co(CO)₄]₂,¹ Hg[Mn(CO)₅]₂,² and those of cyclopentadienyl derivatives of metal carbonyls, such as Hg[π -C₅H₅M(CO)_n]₂,^{3,4} where *n* = 2 or 3 and M = Mo, Cr, or Fe, are known. Although all of these compounds possess three metals covalently bonded, they are all symmetrical, consisting of two identical metal carbonyl portions bonded through mercury. For this reason they are usually referred to as mercury salts. In an extension of our mercury insertion reaction,⁵ we have synthesized what is to the best of our knowledge the first example of a compound containing three different metals covalently bonded.

The new compound, π -cyclopentadienyldicarbonyl-iron mercury cobalt tetracarbonyl, π -C₅H₅Fe(CO)₂-HgCo(CO)₄ (I), was prepared by simply stirring a solution of π -cyclopentadienylmonocarbonyliron μ -dicarbonyl cobalt tricarbonyl⁶ (II) in hexane with mercury under an atmosphere of carbon monoxide at room temperature for 48 hr.



On stirring, the dark red (almost black) solution very gradually changed to dark orange. The orange

(1) W. Hieber and H. Schulten, *Z. anorg. allgem. Chem.*, **232**, 17 (1937).

(2) W. Hieber and W. Schropp, Jr., *Chem. Ber.*, **93**, 455 (1960).

(3) E. O. Fischer, W. Hafner, and H. O. Stahl, *Z. anorg. allgem. Chem.*, **282**, 47 (1955).

(4) E. O. Fischer and R. Böttcher, *Z. Naturforsch.*, **10b**, 600 (1955).

(5) S. V. Digne and M. Orchin, *Inorg. Chem.*, **1**, 965 (1962).

(6) K. K. Joshi and P. L. Pauson, *Z. Naturforsch.*, **17b**, 565 (1962).