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Polyhedron 22 (2003) 3445–3449

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Controlled synthesis of mono-substituted diphosphine iron thiocarboxylate complexes $CpFe(CO)(Ph_2P(CH_2)_nPPh_2)SCOR$ [$n = 1$ (dppm), $n = 2$ (dppe)]. X-ray crystal structure of $CpFe(CO)(dppm\text{-}S)SCO\text{-}3,5\text{-}(NO_2)_2C_6H_3$

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Received 28 May 2003; accepted 21 August 2003

Abstract

Controlled photolytic CO-substitution reaction of the organoiron thiocarboxylate complexes $CpFe(CO)_2SCOR$ ($R = CH_3$, 2-$NO_2C_6H_4$, 4-$NO_2C_6H_4$, 3,5-$(NO_2)_2C_6H_3$) with diphosphines ($Ph_2P(CH_2)_nPPh_2$) [$n = 1$ (dppm), $n = 2$ (dppe)] at 0 °C using 1:1 (metal:ligand) molar ratio afforded exclusively the monosubstituted complexes $CpFe(CO)(Ph_2P(CH_2)_nPPh_2)SCOR$. The complex $CpFe(CO)(Ph_2PCH_2P{=}S(Ph)_2)SCO\text{-}3,5\text{-}(NO_2)_2C_6H_3$ has been prepared and its X-ray structure was determined.

Keywords: Substitution; Iron; Thiocarboxylate; Bisphosphine ligands; Structure

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1. Introduction

Mono- and diphosphine ligands have played a major role in coordination chemistry [1]. In addition to their facile syntheses, their electronic and steric properties can be varied systematically by varying the substituents on the phosphorous and by varying the backbone length of the diphosphines. Photolytic and thermal reactions of the half-sandwich complexes $CpFe(CO)_2X$ ($X =$ halide, alkyl, SiR_3, SnR_3, SR) with EPh_3 ($E = P$, As, Sb) were commonly utilized to prepare CO-substituted complexes. The type of the product formed in these reactions is determined by the nature of X, the type of ER_3, the solvent, the irradiation time, the temperature and the M:L molar ratios. Complexes such as $[CpFe(CO)_2ER_3]X$ ($X =$ halide), $CpFe(CO)(ER_3)COCH_3$, $[CpFe(PR_3)_3]X$ ($X = I$, Br) and $Cp'Fe(CO)(EPh_3)$ SCOR ($Cp' = C_5H_5$, $Bu^tC_5H_4$, 1,3-$Bu^t_2C_5H_3$) are among the important products of such reactions [2–9]. Moreover, diphosphines ($R_2P(CH_2)_nPR_2$) are useful ligands in forming both chelate and/or bridged binuclear complexes depending on the chelating ability of the diphosphine. In forming chelate complexes, the optimum ring size is five, thus $Ph_2P(CH_2)_2PPh_2$ (dppe) is an excellent chelate ligand. $Ph_2PCH_2PPh_2$ (dppm) can chelate but the four membered ring formed is so strained and the ligand has a greater tendency to act as a bridging bidentate ligand. The chelating ability decreases as the chain length increases. A particularly good example is seen in complexes $RhCl(CO)(Ph_2P(CH_2)_nPPh_2)$ which are dimers when $n = 1, 3, 4$ but monomers when $n = 2$ [10,11].

It was recently reported that the photolytic reaction of the alkynyl dicarbonyl derivatives $CpFe(CO)_2(C{\equiv}CR)$ with dppm in THF afforded exclusively the disubstituted

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0277-5387/$ - see front matter
doi:10.1016/j.poly.2003.08.005

derivatives CpFe(dppm)(C≡CR). The "dangling" complexes CpFe(CO)(dppm)(C≡CR) were never isolated but they were detected as an intermediates by ^{31}P NMR [12].

Organoiron thiocarboxylate complexes, Cp′Fe (CO)$_2$ SCOR (Cp′ = C_5H_5, Bu^tC_5H_4, 1,3-Bu$^t_2$$C_5H_3$, R = alkyl, aryl) appear to be interesting systems especially in their behavior towards photolytic CO-substitution reactions with tertiary phosphine ligands. It was found that the photolytic CO-substitution reactions of Cp′Fe(CO)$_2$ SCOR with EPh$_3$ (E = P, As, Sb) afforded exclusively the mono-substituted derivatives Cp′Fe (CO)(EPh$_3$)SCOR irrespective of the reaction conditions [9]. On the other hand, photolytic CO-substitution reactions of CpFe(CO)$_2$SCOR with diphosphine ligands seem to be strongly dependent on the reaction conditions (reaction temperature, the irradiation time, the metal:ligand molar ratio, the backbone length of the diphosphine ligand and the nature of the R group of the thiocarboxylate moiety). The photolytic CO-substitution reaction of CpFe(CO)$_2$SCOR with diphosphine ligands Ph$_2$P(CH$_2$)$_n$PPh$_2$ ($n = 1$–6) afforded the disubstituted derivatives CpFe (Ph$_2$P(CH$_2$)$_n$PPh$_2$)SCOR when $n = 1, 2, 3$ and the monosubstituted derivatives CpFe(CO)(Ph$_2$P (CH$_2$)$_n$PPh$_2$)SCOR when $n = 4, 5, 6$ [13].

In continuation to our efforts in this area, we report here the controlled syntheses of the monosubstituted diphosphine derivatives CpFe(CO)(Ph$_2$P(CH$_2$)$_n$PPh$_2$) SCOR ($n = 1, 2$).

2. Experimental

All reactions were conducted under N$_2$ atmosphere by Schlenk techniques. The organoiron thiocarboxylate complexes CpFe(CO)$_2$SCOR were prepared according to published procedure [14]. Bis(diphenylphosphino)methane (dppm) and bis(diphenylphosphino)ethane (dppe) were purchased from Aldrich and used as received. For column chromatography, silica gel of particle size 0.0063–0.200 mm (70–230 mesh) was employed. All photolytic reactions were carried out using a high-pressure mercury lamp (HANAU, 240–260 nm). Infrared (IR) spectra were recorded on a Nicolet-Impact 410 FT-IR spectrometer and ^{1}H NMR spectra on a Bruker WP 80 SY spectrometer with TMS as internal standard. Elemental analyses were performed by Laboratoire d'Analyse Élémentaire, Université de Montréal, Montréal, Canada. Melting points were measured on an electrothermal melting point apparatus and are uncorrected.

2.1. General procedure for the preparation of CpFe (CO)(Ph$_2$P(CH$_2$)$_n$PPh$_2$)SCOR

A THF solution (100 ml) of the CpFe(CO)$_2$SCOR (1.00 mmol) and the diphosphine (1.0 mmol) were irradiated at 0 °C. The reaction was monitored by IR spectroscopy and irradiation was continued until the disappearance of the two strong terminal carbonyl bands in the ranges 2025–2040 and 1980–1995 cm^{-1} and the appearance of a new band in the range 1940–1970 cm^{-1}. The solvent was removed under vacuum and the residue was dissolved in a minimum amount of CH$_2$Cl$_2$ and transferred to a silica gel column made up in hexane. Elution with hexane removes the excess diphosphine ligand. The red band of the product was eluted with CH$_2$Cl$_2$/hexane solution (1:1 volume ratio). Evaporation of the solvent under vacuum and recrystallization from CH$_2$Cl$_2$–hexane mixture afforded the monosubstituted complexes CpFe(CO)(Ph$_2$P(CH$_2$)$_n$ PPh$_2$) SCOR (**1**–**4**).

*2.1.1. CpFe(CO)(Ph$_2$PCH$_2$PPh$_2$)SCO-3,5-(NO$_2$)$_2$C$_6$H$_3$ (**1a**)*

Yield = 50%. m.p. = 138–140 °C. ^{1}H NMR (CDCl$_3$): δ 3.50 (s, 2H, CH$_2$); 4.43 (s, 5H, Cp); 7.25 (m, 20H, 2PPh$_2$); 9.40 (m, 3H, Ph). IR (KBr, cm^{-1}) $\nu_{C\equiv O}$, 1959 (vs); $\nu_{C=O}$, 1600 (m); ν_{NO_2}, 1531 (s), 1346 (s). *Anal.* Calc. for $C_{38}H_{30}FeN_2O_6P_2S$: C, 60.14; H, 3.95; N, 3.68; S, 4.21. Found C, 59.85; H, 3.95; N, 3.76; S, 4.33%.

*2.1.2. CpFe(CO)(Ph$_2$P(CH$_2$)$_2$PPh$_2$)SCO-3,5-(NO$_2$)$_2$C$_6$H$_3$ (**1b**)*

Yield = 65%. m.p. = 97–99 °C. ^{1}H NMR (CDCl$_3$): δ 2.79 (m, 4H, CH$_2$); 4.49 (s, 5H, Cp); 7.29 (s, 20H, 2PPh$_2$); 9.17 (m, 3H, Ph). IR (KBr, cm^{-1}): $\nu_{C\equiv O}$, 1959 (vs); $\nu_{C=O}$, 1604 (m); ν_{NO_2}, 1537, 1341 (s). *Anal.* Calc. for $C_{39}H_{32}FeN_2O_6P_2S$: C, 60.48; H, 4.16; N, 3.62; S, 4.14. Found C, 60.46; H, 4.11; N, 3.49; S, 4.28%.

*2.1.3. CpFe(CO)(PPh$_2$CH$_2$PPh$_2$)SCO-4-NO$_2$C$_6$H$_4$ (**2a**)*

Yield = 40%. m.p. = 120–122 °C. ^{1}H NMR (CDCl$_3$): δ 3.51 (s, 2H, CH$_2$); 4.52 (s, 5H, Cp); 7.25 (m, 20H, 2PPh$_2$); 8.10 (dd, 4H, Ph). IR (KBr, cm^{-1}): $\nu_{C\equiv O}$, 1963 (vs); $\nu_{C=O}$, 1586 (s); ν_{NO_2}, 1516, 1335 (s). *Anal.* Calc. for $C_{38}H_{31}FeNO_4P_2S$: C, 63.79; H, 4.37; N, 1.96; S, 4.48. Found C, 64.10; H, 4.31; N, 1.88; S, 4.62%.

*2.1.4. CpFe(CO)(Ph$_2$P(CH$_2$)$_2$PPh$_2$)SCO-4-NO$_2$C$_6$H$_4$ (**2b**)*

Yield = 70%. m.p. = 201–203 °C. ^{1}H NMR (CDCl$_3$): δ 2.55 (t, 4H, CH$_2$); 4.47 (s, 5H, Cp); 7.23 (m, 20H, 2PPh$_2$); 8.28 (dd, 4H, Ph). IR (KBr, cm^{-1}): $\nu_{C\equiv O}$, 1955 (vs); $\nu_{C=O}$, 1606 (m); ν_{NO_2}, 1521, 1349 (s). *Anal.* Calc. for $C_{39}H_{33}FeNO_4P_2S$: C, 64.21; H, 4.56; N, 1.92; S, 4.40. Found C, 64.85; H, 4.58; N, 2.01; S, 4.32%.

*2.1.5. CpFe(CO)(Ph$_2$P(CH$_2$)$_2$PPh$_2$)SCO-2-NO$_2$C$_6$H$_4$ (**3b**)*

Yield = 62%. m.p. = 185–187 °C. ^{1}H NMR (CDCl$_3$): δ 2.56 (t, 4H, CH$_2$); 4.56 (s, 5H, Cp); 7.43 (m, 20H, 2PPh$_2$); 7.80 (dd, 4H, Ph). IR (KBr, cm^{-1}): $\nu_{C\equiv O}$, 1950 (vs); $\nu_{C=O}$, 1605 (m); ν_{NO_2}, 1530, 1340 (s). *Anal.* Calc. for

$C_{39}H_{33}FeNO_4P_2S$: C, 64.21; H, 4.56; N, 1.92; S, 4.40. Found C, 64.56; H, 4.61; N, 1.98; S, 4.71%.

2.1.6. *CpFe(CO)(PPh₂P(CH₂)₂PPh₂)SCOCH₃* (**4b**)

Yield = 55%. m.p. = 212–214 °C. ^{1}H NMR ($CDCl_3$): δ 1.42 (s, 3H, CH_3); 2.72 (t, 4H, CH_2); 4.58 (s, 5H, Cp); 7.50 (m, 20H, $2PPh_2$). IR (KBr, cm^{-1}): $\nu_{C\equiv O}$, 1950 (vs); $\nu_{C=O}$, 1610 (m). *Anal.* Calc. for $C_{34}H_{32}FeO_2P_2S$: C, 65.60; H, 5.18; S, 5.15. Found C, 65.34; H, 5.22; S, 5.37%.

2.2. *Preparation of CpFe(CO)(Ph₂PCH₂P=S(Ph)₂)S-CO-3,5-(NO₂)₂C₆H₃* (**1c**)

A methylene chloride solution containing **1a** (2.0 g, 1.5 mmol) and (μ-S_3)[CpFe$(CO)_2$]$_2$ (1.1 g, 0.5 mmol) was stirred at room temperature for 2 h. The solvent was evaporated under vacuum and the residue was introduced to a chromatography column made up in hexane. A red band of [Fp–Fp] was eluted (CH_2Cl_2/hexane) (3:7). The product **1c** was eluted with (CH_2Cl_2/hexane) (7:3). Recrystallization from $CHCl_3$/hexane afforded a dark-red crystals. Yield = 80%. m.p. = 110–112 °C. ^{1}H NMR ($CDCl_3$): δ 3.21 (s, 2H, CH_2); 4.45 (s, 5H, Cp); 7.23 (m, 20H, $2PPh_2$); 9.38 (m, 3H, Ph). IR (KBr, cm^{-1}): $\nu_{C\equiv O}$, 1960 (vs); $\nu_{C=O}$, 1600 (s); ν_{NO_2}, 1534, 1345 (s). *Anal.* Calc. for $C_{38}H_{30}FeN_2O_6P_2S_2 \cdot CHCl_3$: C, 51.34; H, 3.40; N, 3.07; S, 7.03. Found C, 51.19; H, 3.52; N, 3.17; S, 7.08%.

2.3. *Preparation of the bridging complex (μ-Ph₂P(CH₂)₂PPh₂)[CpFe(CO)SCO-3,5-(NO₂)₂C₆H₃]₂* (**5b**)

In a procedure similar to that described for **1**, solution of CpFe$(CO)_2$SCO-3,5-C_6H_3 $(NO_2)_2$ (2.47 g, 1.0 mmol) and $Ph_2P(CH_2)_2PPh_2$ (1.24 g, 0.5 mmol) in THF was irradiated for 30 min. Column chromatography afforded a dark-red band, which was eluted with CH_2Cl_2/hexane (9:1). Removal of the solvent under vacuum and recrystalization from CH_2Cl_2/hexane afford red crystals of the title compound. Yield = 50%. m.p. = 126–128 °C. ^{1}H NMR (CD_2Cl_2): δ 2.58 (s, 4H, CH_2); 4.50 (s, 10H, Cp); 7.50 (m, 26H, $2PPh_2 + C_6H_4$). IR (KBr, cm^{-1}): $\nu_{C\equiv O}$, 1948(vs); $\nu_{C=O}$, 1601 (m); ν_{NO_2}, 1534, 1437(s). *Anal.* Calc. for ($C_{26}H_{30}FeN_2O_6PS$): C, 50.66; H, 4.91; N, 4.54; S, 5.20. Found C, 49.79; H, 4.57; N, 4.43; S, 5.42%.

2.4. *Crystal structural analysis for CpFe(CO)(Ph₂PCH₂P(=S)Ph₂)SCO-3,5-(NO₂)₂C₆H₃* (**1c**)

Crystals suitable for the X-ray study were obtained by recrystallization from $CHCl_3$. Compound **1c** ($C_{38}H_{30}N_2O_6P_2S_2Fe$), molar mass 782.55; crystallizes in the monoclinic system, space group $P2_1/n$ with $a = 12.860(3)$ Å, $b = 11.052(2)$ Å, $c = 29.955(6)$ Å and $\beta = 98.667(5)°$; $V = 4208.8(15)$ Å^3; $d_{calc.} = 1.251$ g/cm^3, $Z = 4$; $\mu = 0.576$ mm^{-1}.

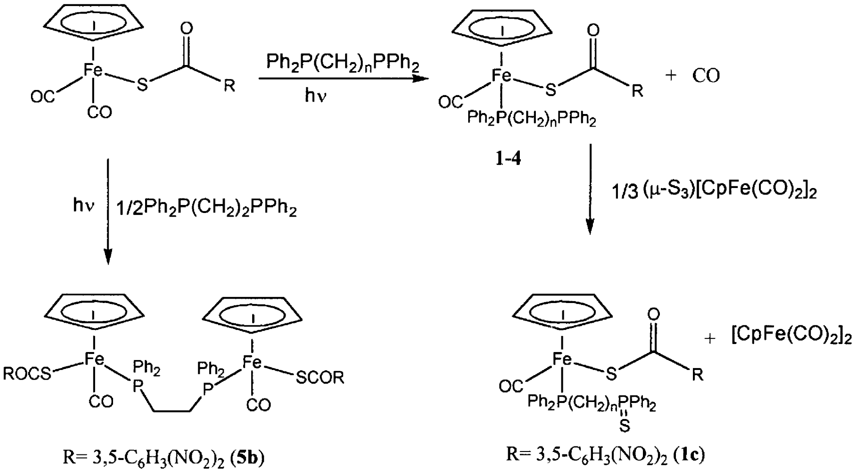
The cell constants and reflections were measured at a temperature of 173(2) K on a Bruker Smart 1K CCD detector with a graphite monochromator, λ(Mo Kα) = 0.71073 Å. An ω scan with a scan range $1.65° \leqslant 2\theta \leqslant 29.26°$ has been employed. There were 3947 independent significant reflections ($I \geqslant 2\sigma$). The structure was solved by use of the program SHELX 97 [15] by direct methods. The position of disordered or not refinable solvent molecule was calculated by the PLATON [16] integrated SQEEZE [17] procedure and the corresponding electron density was subtracted before the final refinement. All non-hydrogen atoms were refined with anisotropic thermal parameters. Hydrogen atoms were placed in the calculated positions. The refinement converged at $R_1 = 0.0958$ and $R_w = 0.1940$.

3. Results and discussion

In our study of the photolytic CO-substitution reaction of CpFe$(CO)_2$SCOR with diphosphine ligands we observed that the reaction temperature and the irradiation time are significant in determining the type of product formed [13]. Thus the monosubstituted derivatives, CpFe(CO)($Ph_2P(CH_2)_nPPh_2$)SCOR ($n = 1, 2$), could be prepared if the reaction temperature is kept at 0 °C and the irradiation time is stopped when the first CO group is substituted (Scheme 1). Small amount (ca. 5–10%) of a red complexes have been obtained from these reactions. These complexes were found to be the dimers of the formula (μ-$Ph_2P(CH_2)_nPPh_2$)[CpFe(CO)SCOR]$_2$. The yield of these products can be maximized by doing a separate reaction using 2:1 (metal:ligand) ratio. The complex (μ-Ph_2P $(CH_2)_2PPh_2$)[CpFe(CO)SCO-3,5-$C_6H_3(NO_2)_2$]$_2$ has been prepared and characterized by elemental analysis and spectral data.

The monosubstituted derivatives (**1**–**4**) which are stable in the solid state seem to be novel complexes in view of the large chelation tendency of such diphosphine ligands and in view of the unsuccessful attempts to isolate them in similar systems as CpFe$(CO)_2$(C≡CR) [12]. Moreover, these newly prepared complexes are of special interest, because they can be considered as a good precursors for the synthesis of homo and hetero phosphino-bridged binuclear complexes in which the two metals are in close proximity that promote catalytic organometallic reactions.

Complexes **1**–**4** were characterized by elemental analysis, IR and ^{1}H NMR spectral data. Their IR spectra exhibit one very strong band in the range 1940–1970 cm^{-1} corresponding to the carbonyl group bon-



For **1-4**: n= 1 (**a**), 2 (**b**); R= 3,5-$C_6H_3(NO_2)_2$ (**1**), 2-$C_6H_4NO_2$ (**2**), 4-$C_6H_4NO_2$ (**3**), CH_3 (**4**)

Scheme 1.

ded to the iron atom. The C=O stretching band of the thiocarboxylate moiety appears in the range 1586–1621 cm^{-1}. The assignment of these bands was made on the bases of the reported results of the organoiron thiocarboxylate complexes [14]. Their ^1H NMR spectra show the characteristic protons in their expected chemical shift regions.

In an attempt to test the reactivity of the unbonded phosphine of the prepared mono-substituted complexes, the complex $CpFe(CO)(Ph_2PCH_2PPh_2)SCO\text{-}3,5\text{-}C_6H_3(NO_2)_2$, **1a**, was treated with $(\mu\text{-}S_3)[CpFe\ CO)_2]_2$, which is known to undergo smooth desulfurization with tertiary phosphines [18]. Indeed, desulfurization took place and the unbonded phosphine abstracted sulfur forming the phosphine–sulfide derivative $CpFe(CO)(Ph_2PCH_2P{=}S(Ph)_2)SCO\text{-}3,5\text{-}(NO_2)_2C_6H_3$, **1c**. The structure of **1c** was determined by X-ray analysis.

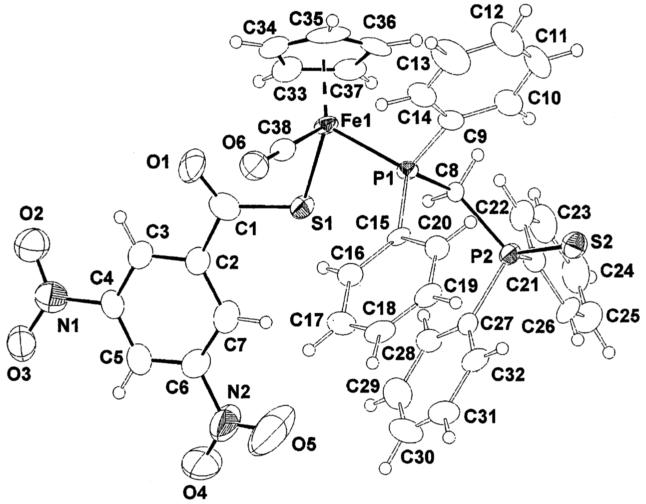


Fig. 1. Selected bond lengths (Å) and angles (°): Fe–C38 = 1.755(6), Fe–C33 = 2.102(7), Fe–C34 = 2.085(7), Fe–C35 = 2.058(7), Fe–C36 = 2.107(7), Fe–C37 = 2.089(6), Fe–P1 = 2.2092(18), Fe–S1 = 2.273(2), P2–S2 = 1.951(3), S1–C1 = 1.760(8), C1–O1 = 1.215(9); C38–Fe–P1 = 93.5(2), C38–Fe–S1 = 93.5(2), C1–S1–Fe = 106.2(3), O1–C1–S1 = 125.7(6), P2–C8–P1 = 124.6(3).

3.1. *Description of the crystal structure of $CpFe(CO)(Ph_2PCH_2P{=}S(Ph_2)SCO\text{-}3,5\text{-}(NO_2)_2C_6H_3$ (1c)*

The complex consists of a monomeric molecule in which the Fe-atom is bonded to four different groups (Fig. 1). The cyclopentadienyl ring is bound to the Fe-atom in a η^5-fashion with Fe–C bond distances ranging between 2.058(7) and 2.107(7) Å. The (dppm-S) ligand acts as monodentate ligand through the P-atom with an Fe–P bond distance of 2.209(2) Å. The Fe–S bond distance of the thiocarboxylate moiety is 2.273(3), which is in agreement with that of the Fe–S thiocarboxylate complexes [14].

The coordination around the Fe-atom of the cyclopentadienyl ring (taken as bound to its centroid), the CO group, the thiocarboxylate moiety and the dppm-S ligand can be described as a three-legged piano stool. Moreover, the values of the angles involving the three legs C_{38}–Fe–S_1 93.5(2)°, C_{38}–Fe–P_1 93.5(2)° and S_1–Fe–P_1 92.16(7)° are almost equal.

4. Supplementary data

Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Data Center, CCDC No. 209309. Copies of the information may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge, CB2 1EZ, UK (fax: +44-1223-336033; e-mail: deposit@ccdc.cam.ac.uk or www: http//www.ccdc.cam.ac.uk).

Acknowledgements

The authors thank the Deanship of Research, Jordan University of Science & Technology for financial support, Grant No. 143/99.

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