SYNTHESIS AND ANTIMICROBIAL ACTIVITY OF SULPHONAMIDE, IMIDAZOLINONE, N,N'-(DIARYLDIAMINO)PHOSPHINIC CHLORIDE DERIVATIVES HAVING "FLUCHLORALIN" MOIETY.

D.M.Purohit and V. H. Shah*

Department of Chemistry, Saurashtra University, Rajkot - 360 005. India

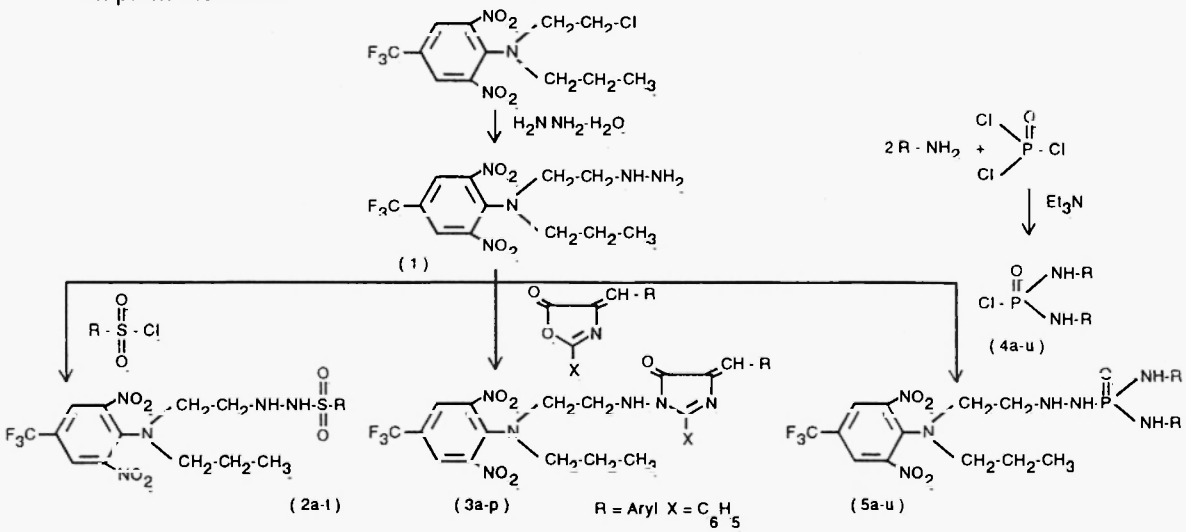
ABSTRACT :

Some newly synthesised compounds of 2,6-dinitro-4-(trifluoromethyl)-N-(arylsulphonylhydrazinoethyl)-N-(propyl)-aniline(2a-t),1-N'-(propyl)-2',6'-dinitro-4'-(trifluoromethyl)-phenylamino-N'-(ethylamino)-4-arylidene-2-phenyl-5-imidazolinones(3a-p),N,N'-(diaryldiamino)phosphinic chlorides (4a-u), N,N'-(diaryldiamino) phosphonic hydrazinoethyl-N-(propyl)-2,6-dinitro-4-(trifluoromethyl)-aniline(5a-u) consisting of fluchloralin moiety. The synthesised compounds are screened for their antimicrobial activities. The products (2a-t, 3a-p, 4a-u, 5a-u) have been elucidated by elemental analyses and spectral (IR , ^{1}HNMR)data.

INTRODUCTION :

Fluchloralin is a well known agrochemical for their herbicidal[1-3] properties, while worked into soil for preplant and pre-emergence control of annual grasses and some broad-leaf weeds in cotton and soyabeans.

In view of getting potent agrochemical agents, sulphonamides imidazolinones , organo phosphorus linkages have been introduced in fluchloralin as a part of research study. sulphonamides[4-6] , 5-imidazolinones [7-9], N,N'-(diaryldiamino)phosphinic chlorides[10] derivatives.Organophosphorus compounds are known for their pesticidal[11] and fungicidal[12] action on plant diseases.



2,6-dinitro-4-trifluoromethyl-N-(1'-hydrazinoethyl)-N-(propyl)-aniline-(1) have been prepared by 2,6-dinitro-4-(trifluoromethyl)-N- (2-chloroethyl)N(propyl)-aniline(fluchloralin) with hydrazine hydrate. 2,6-dinitro-4-(trifluoromethyl)-N-(arylsulphonyl hydrazinoethyl)-N-(propyl)-aniline (2a-t) have been synthesised by the reaction of arylsulphonyl chlorides with compound-(1). Compounds (3a-p) have been synthesised by the condensation of compound(1) with preformed azlactones prepared by Erlenmeyer reaction [13].N,N'-(diaryldiamino)phosphinic chlorides(4a-u) have been synthesised by the action of two moles of arylamines and one mole of

Table - I : The physical data and antimicrobial activity of compounds [2a-t,3a-p,4a-u,5a-u]

Compd	R	Molecular formula	M.P. oC	Yield %	Nitrogen (%)		Antibacterial activity Zone of inhibition in m.m.			Antifungal activity Zone of inhibition in m m.	
					Calcd	Found	B.mega	B.subtilis	E.Coli	A aragen	A.awamori
2a	C_6H_5	$C_{18}H_{20}O_6N_5SF_3$	58	80.16	14.25	14.21	17	18	15	20	20
2b	3-COOH.C_6H_4	$C_{19}H_{20}O_8N_5SF_3$	71	76.83	13.08	13.05	19	17	17	19	21
2c	4-CH=CH.COOH.C_6H_4	$C_{21}H_{22}O_8N_5SF_3$	256	77.81	12.47	12.43	21	18	16	20	20
2d	3-COOH,-4-OH.C_6H_3	$C_{19}H_{20}O_9N_5SF_3$	233	79.03	12.70	12.68	21	19	19	24	24
2e	4-$OCH_2COOH.C_6H_4$	$C_{20}H_{22}O_9N_5SF_3$	84	71.36	12.38	12.35	18	15	17	20	22
2f	3-COOH-4-$OCH_3C_6H_3$	$C_{20}H_{22}O_9N_5SF_3$	192	72.52	12.38	12.34	20	20	22	25	25
2g	3-COOH,-5-$OCH_3C_6H_3$	$C_{20}H_{22}O_9N_5SF_3$	183	71.87	12.38	12.36	22	19	20	19	21
2h	3-COOH,6-$OCH_3C_6H_3$	$C_{20}H_{22}O_9N_5SF_3$	169	73.31	12.38	12.35	22	19	19	20	20
2i	3-COOH,4-$CH_3C_6H_3$	$C_{20}H_{22}O_8N_5SF_3$	48	79.35	12.75	12.72	19	17	17	18	24
2j	3-COOH,5-$CH_3C_6H_3$	$C_{20}H_{22}O_8N_5SF_3$	69	78.67	12.75	12.73	18	16	18	17	22
2k	3-COOH,6-$CH_3C_6H_3$	$C_{20}H_{22}O_8N_5SF_3$	105	82.97	12.75	12.71	17	18	19	20	20
2l	3-COOH,4-Cl.C_6H_3	$C_{19}H_{19}O_8N_5SF_3Cl$	174	81.36	12.29	12.26	22	20	21	23	24
2m	3-COOH,5-Cl.C_6H_3	$C_{19}H_{19}O_8N_5SF_3Cl$	158	79.14	12.29	12.25	24	18	23	21	21
2n	3-COOH,6-Cl.C_6H_3	$C_{19}H_{19}O_8N_5SF_3Cl$	167	78.48	12.29	12.24	21	17	19	20	22
2o	4-Cl.C_6H_4	$C_{18}H_{19}O_6N_5SF_3Cl$	96	75.12	13.32	13.29	19	20	18	24	20
2p	4-Br.C_6H_4	$C_{18}H_{19}O_6N_5SF_3Br$	118	76.01	12.28	12.25	18	16	17	20	19
2q	2,5-$(Cl)_2.C_6H_3$	$C_{18}H_{18}O_6N_5SF_3Cl_2$	65	71.08	12.50	12.46	23	18	22	23	20
2r	2,5-$(Br)_2.C_6H_3$	$C_{18}H_{18}O_6N_5SF_3Br_2$	59	78.72	10.78	10.75	20	17	19	18	19
2s	4-$NHCOCH_3.C_6H_4$	$C_{20}H_{23}O_7N_6SF_3$	147	73.00	15.32	15.30	18	18	15	17	17
2t	4-$NHCOCH_3$,3-COOH.C_6H_3	$C_{21}H_{23}O_9N_6SF_3$	134	77.96	14.18	14.16	17	16	15	19	19
3a	C_6H_5	$C_{28}H_{25}O_5N_6F_3$	80	70.13	14.43	14.40	18	17	16	20	20
3b	2-Cl.C_6H_4	$C_{28}H_{24}O_5N_6F_3Cl$	171	63.98	13.62	13.58	20	16	24	26	21
3c	4-Cl.C_6H_4	$C_{28}H_{24}O_5N_6F_3Cl$	109	67.07	13.62	13.59	21	17	25	24	23
3d	2-$NO_2.C_6H_4$	$C_{28}H_{24}O_7N_7F_3$	205	72.36	15.62	15.59	19	14	23	21	19
3e	3-$NO_2C_6H_4$	$C_{28}H_{24}O_7N_7F_3$	120	65.50	15.62	15.61	20	15	22	20	18
3f	4-$NH_2C_6H_4$	$C_{28}H_{26}O_5N_7F_3$	132	62.87	16.41	16.38	17	13	20	18	17
3g	4-$OCH_3C_6H_4$	$C_{29}H_{27}O_6N_6F_3$	190	59.17	13.72	13.70	18	12	18	17	19
3h	2-OH.C_6H_4	$C_{28}H_{25}O_6N_6F_3$	52	68.29	14.04	14.01	19	14	19	19	20
3i	4-OH.C_6H_4	$C_{28}H_{25}O_6N_6F_3$	138	75.78	14.04	14.00	21	18	18	21	23
3j	C_4H_3O	$C_{27}H_{23}O_6N_6F_3$	135	59.14	14.38	14.35	16	11	17	18	19
3k	4-$N,N(CH_3)_2C_6H_4$	$C_{30}H_{30}O_5N_7F_3$	123	76.49	15.68	15.64	18	12	16	20	17
3l	C_6H_5CH=CH-	$C_{30}H_{27}O_5N_6F_3$	148	71.30	13.81	13.80	20	12	19	15	18
3m	4-$SCH_3C_6H_4$	$C_{29}H_{27}O_5N_6SF_3$	220	58.75	13.37	13.35	22	13	21	19	20
3n	4-OH,3-$OCH_3C_6H_3$	$C_{29}H_{27}O_7N_6F_3$	184	69.02	13.37	13.34	21	14	22	20	20
3o	3:4:5(-$OCH_3)_3C_6H_2$	$C_{31}H_{31}O_8N_6F_3$	141	67.92	12.20	12.17	19	15	23	21	24
3p	3:4(-$OCH_3)_2C_6H_3$	$C_{30}H_{29}O_7N_6F_3$	126	76.48	13.08	13.06	18	14	21	19	17
4a	C_6H_5	$C_{12}H_{12}ON_2PCl$	200	73.16	10.51	10.42	18	12	13	17	15

4b	2-Cl.C_6H_4	$C_{12}H_{10}ON_2PCl_3$	216	74.03	8.34	8.30	20	15	17	20	17
4c	3-Cl.C_6H_4	$C_{12}H_{10}ON_2PCl_3$	194	80.98	8.34	8.28	21	14	19	19	16
4d	4-Cl.C_6H_4	$C_{12}H_{10}ON_2PCl_3$	183	76.00	8.34	8.31	19	16	20	18	23
4e	2,4-$(Cl)_2$.C_6H_3	$C_{12}H_8ON_2PCl_5$	98	68.18	6.92	6.90	20	18	21	17	19
4f	2,6-$(Cl)_2$.C_6H_3	$C_{12}H_8ON_2PCl_5$	113	69.37	6.92	6.88	24	17	18	26	20
4g	3,4-$(Cl)_2$.C_6H_3	$C_{12}H_8ON_2PCl_5$	84	70.15	6.92	6.87	22	19	25	24	21
4h	2,6-$(Cl)_2$.4$NO_2C_6H_2$	$C_{12}H_6O_5N_4PCl_5$	180	78.87	11.32	11.28	24	19	26	23	24
4i	2-OCH_3.C_6H_4	$C_{14}H_{16}O_3N_2PCl$	222	72.58	8.57	8.53	18	16	17	16	20
4j	3-OCH_3.C_6H_4	$C_{14}H_{16}O_3N_2PCl$	211	74.42	8.57	8.54	17	17	19	18	19
4k	4-OCH_3.C_6H_4	$C_{14}H_{16}O_3N_2PCl$	186	75.86	8.57	8.53	19	15	16	19	17
4l	2-CH_3.C_6H_4	$C_{14}H_{16}ON_2PCl$	167	70.92	9.51	9.45	14	13	15	16	15
4m	3-CH_3.C_6H_4	$C_{14}H_{16}ON_2PCl$	156	69.33	9.51	9.48	15	12	14	18	18
4n	4-CH_3.C_6H_4	$C_{14}H_{16}ON_2PCl$	174	68.48	9.51	9.49	17	14	18	19	16
4o	2-NO_2.C_6H_4	$C_{12}H_{10}O_5N_4PCl$	238	81.08	15.71	15.65	19	13	19	17	18
4p	3-NO_2.C_6H_4	$C_{12}H_{10}O_5N_4PCl$	95	82.32	15.71	15.63	20	15	17	18	24
4q	4-NO_2.C_6H_4	$C_{12}H_{10}O_5N_4PCl$	151	80.49	15.71	15.68	18	17	20	20	21
4r	3-Cl, 6-CH_3.C_6H_3	$C_{14}H_{14}ON_2PCl_3$	188	73.50	7.70	7.67	17	16	18	26	20
4s	4-Cl, 3-NO_2.C_6H_3	$C_{14}H_{14}O_5N_4PCl$	104	79.28	14.56	14.50	14	13	15	22	18
4t	2-CH_3,5-NO_2.C_6H_3	$C_{14}H_{14}O_5N_4PCl$	129	74.03	14.56	14.49	16	12	17	20	15
4u	2-CH_3,3-NO_2.C_6H_3	$C_{14}H_{14}O_5N_4PCl$	203	76.49	14.56	14.52	18	14	15	19	16
5a	C_6H_5	$C_{24}H_{27}O_5N_7PF_3$	47	74.04	16.87	16.85	16	14	13	15	17
5b	2-Cl.C_6H_4	$C_{24}H_{25}O_5N_7PF_3Cl_2$	173	71.86	15.07	15.00	18	16	15	14	14
5c	3-Cl.C_6H_4	$C_{24}H_{25}O_5N_7PF_3Cl_2$	146	72.35	15.07	15.04	20	21	17	19	20
5d	4-Cl.C_6H_4	$C_{24}H_{25}O_5N_7PF_3Cl_2$	130	76.12	15.07	15.04	23	18	16	20	24
5e	2,4-$(Cl)_2$.C_6H_3	$C_{24}H_{23}O_5N_7PF_3Cl_4$	101	80.44	13.63	13.60	21	17	21	26	21
5f	2,6-$(Cl)_2$.C_6H_3	$C_{24}H_{23}O_5N_7PF_3Cl_4$	125	81.82	13.63	13.58	20	22	23	24	23
5g	3,4-$(Cl)_2$.C_6H_3	$C_{24}H_{23}O_5N_7PF_3Cl_4$	78	69.77	13.63	13.55	24	18	26	28	22
5h	2,6-$(Cl)_2$.4$NO_2C_6H_2$	$C_{24}H_{21}O_9N_9PF_3Cl_4$	135	83.64	15.57	15.50	21	17	24	23	26
5i	2-OCH_3.C_6H_4	$C_{26}H_{31}O_7N_7PF_3$	62	71.82	15.29	15.23	18	14	20	18	20
5j	3-OCH_3.C_6H_4	$C_{26}H_{31}O_7N_7PF_3$	145	73.46	15.29	15.20	19	13	19	17	19
5k	4-OCH_3.C_6H_4	$C_{26}H_{31}O_7N_7PF_3$	160	76.41	15.29	15.25	21	15	17	19	18
5l	2-CH_3.C_6H_4	$C_{26}H_{31}O_5N_7PF_3$	165	69.08	16.09	16.03	17	12	18	16	17
5m	3-CH_3 C_6H_4	$C_{26}H_{31}O_5N_7PF_3$	158	72.14	16.09	16.01	16	14	15	15	16
5n	4-CH_3.C_6H_4	$C_{26}H_{31}O_5N_7PF_3$	141	73.38	16.09	16.04	15	13	14	17	12
5o	2-NO_2.C_6H_4	$C_{24}H_{25}O_9N_9PF_3$	52	77.54	18.78	18.73	18	18	20	19	18
5p	3-NO_2.C_6H_4	$C_{24}H_{25}O_9N_9PF_3$	71	75.92	18.78	18.71	20	14	19	18	17
5q	4-NO_2.C_6H_4	$C_{24}H_{25}O_9N_9PF_3$	104	74.08	18.78	18.70	17	15	21	17	19
5r	3-Cl, 6-CH_3.C_6H_3	$C_{26}H_{29}O_5N_7PF_3Cl_2$	111	80.47	14.45	14.40	23	17	18	25	20
5s	4-Cl, 3-NO_2.C_6H_3	$C_{26}H_{28}O_9N_9PF_3$	89	70.14	18.05	18.03	20	16	17	18	18
5t	2-CH_3,5-NO_2.C_6H_3	$C_{26}H_{28}O_9N_9PF_3$	113	72.76	18.05	18.00	19	15	16	19	17
5u	2-CH_3,3-NO_2.C_6H_3	$C_{26}H_{28}O_9N_9PF_3$	97	69.93	18.05	18.02	21	13	15	20	19

phosphorusoxychloride in presence of triethyl amine as basic catalyst to literature method[14]. N,N'-(Diaryldiamino) phosphonic hydrazinoethyl-N-(propyl)-2,6-dinitro-4-(trifluoromethyl)-aniline (5a-u) have been prepared by the condensation of compound -(1) with N,N'-(diaryldiamino)phosphinic chlorides in presence of triethylamine.

The constitution of the products have been delineated by spectral data. The products have been evaluated for their antimicrobial screening.

PHYSICAL DATA AND ANTIMICROBIAL ACTIVITY :

Sulphonamides (2a-t), imidazolines(3a-p) organophosphorous derivatives (4a-u,5a-u) were evaluated in vitro for antibacterial against, **B.mega, B.Subtilis, E.Coli, A.arogens** and for antifungal activity against **A.awamori** using DMF as solvent at 50 μg concentration by cup-plate method[15]. After 24 hrs. of incubation at 37°C, the zone of inhibition were measured in m.m.. The activity was compared with the known antibiotics, viz. Ampicillin, Chloramphenicol, Norfloxacin, Gresiofluvin at same concentration which is represented in Table - I.

RESULTS AND DISCUSSION :

In the case of Antimicrobical activity of all synthesised compounds exhibited moderate to good remarkable and comparable activity with known choosen standerd drugs at same concentration which is represented in Table -II.

Table - II :

ANTIMICROBIAL ACTIVITY :
Conclusion
Maximum Antimicrobial activity

Compounds data	B.mega	B.substilis	E.coli	A.arogens	A. awamori
(2a-t)	2g,2h,2k,2l, 2q	2a,2c,2d,2f 2g,2h.2k,2l 2m,2o,2q,2s	2d,2f,2g,2h,2k, 2k,2l,2m,2n,2q, 2r	2d,2f,2l,2m 2o,2q	2d,2f,2i,2l
(3a-p)	3m	3i	3b,3c,3d,3e,3f 3h,3l,3m,3n,3o, 3p	3b,3c,3d,3i 3o	3c,3o
(4a-u)	4f,4h	4e,4g,4h	4c,4d,4e,4g,4h, 4j,4o.4q	4f,4g,4h,4q 4r,4s,4t	4d,4h,4p
(5a-u)	5d,5g,5t	5c,5d,5f,5g,5o	5e,5f,5g,5h,5i 5j,5o,5p,5q	5e,5f,5g,5h,5r	5d,5f,5h
Comparable activity with known standard drugs					
1. Ampicillin (50 mg)	22	18	17	21	-
2. Chloramphenicol (50 mg)	24	19	19	26	-
3. Norfloxacin (50 mg)	24	19	25	27	-
4. Griseofulvin (50 mg)	-	-	-	-	23

EXPERIMENTAL :

All the melting points were taken in open capillaries and are uncorrected. IR absorption spectra (υin cm^{-1}) were recorded on a shimadzu IR-435 spectrophotometer using KBr pellet and 1HNMR spectra on BRUKER-spectrometer (300MHz) using DMSO as internal standard. purity of the compounds were routinely checked by TLC using silica Gel G.

2-6-Dinitro-4-(trifluoromethyl)-N-(1'-hydrazinoethyl)-N-(propyl)-aniline - (1) :

A mixture of 2,6-dinitro-4-(trifluoromethyl)-N-(2-chloroethyl)-N-(propyl)-aniline (3.55 g, 0.01 mole),hydrazinehydrate (0.75g, 0.15 mole) in pyridine and ethanol (10 ml) was refluxed for 4 hrs . The products were cooled and poured into crused ice filtred, dried and recrystallised from ethanol. m.p. 59°C, Yield : 85.00% (Found : C, 41.01; H, 4.49; N, 19.87; $C_{12}H_{16}O_4N_5F_3$ requires : C, 41.02; H, 4.55; N, 19.94 %) IR (KBr) : 3300 (-NH Str.), 2965 (C-H str.), 1580 (C-N), 1485 (N=O,R-NO_2 str.),1250 (C-F str.) ^{1}HNMR (TFA) : 0.91-1.02 (-N-CH_2-CH_2-CH_3), 3.41-3.43 (-N-CH_2-CH_2-), 8.06(Ar-H) 8.4 (-NH-NH_2).

2-6-Dinitro-4-(trifluoromethyl)-N-(3'-carboxy-4'-methoxyphenyl sulphonylhydrazinoethyl)-N-(propyl)-aniline - (2i) :

A mixture of compound -(1) (3.51g, 0.01 mole),3-carboxy-4-methoxy benzene sulphonyl chloride (2.50g, 0.01 mole) in pyridine (0.5 ml) and ethanol (20 ml) was refluxed for 120°C temp. The products was cooled and poured into crused ice containing enough HCl to neutralise pyridine. The product was filtred, washed with water, dried and recrystallised from ethanol. m.p. 48°C, Yield : 79.35% (Found : C, 43.65; H, 3.98 ; N, 12.72 ; $C_{20}H_{22}O_8N_5SF_3$ requires : C, 43.71; H, 4.00; N, 12.75 %) IR (KBr) : 3300-3240 (N-H Str.), 2950 (C-H str.), 1640 (C=O), 1485 (-N=O,R-NO_2 str.),1310 (S=O str.) 1000-1240 cm^{-1}(C-F str.) ^{1}HNMR (DMSO) :δ 0.86-0.91 (-N-CH_2-CH_2-CH_3),1.54- 1.67(-N-CH_2-CH_2-CH_3) , 2.99 - 3.04 (-N-CH_2-CH_2-CH_3)3.34-3.38 (-N-CH_2-CH_2-), 3.62 - 3.67 -(N-CH_2-CH_2-) 3.85 (-OCH_3) 6.7-8.1 (Ar-H).

Similarly others compounds (2a-t) were prepared and their physical data are recorded in table - I.

1-N'-propyl-2',6'-dinitro-4'-(trifluoromethyl)-phenylamino-N'-(ethyl amino)-4- (3'',4''-dimethoxy benzylidine)-2-phenyl-5-imidazolinone - (3p) :

A mixture of compound -(1) (3.51g, 0.01 mole),4-(3',4'-dimethoxybenzylidine)-2-phenyl-5-oxazolone (3.09g, 0.01 mole) in pyridine (10 ml) was refluxed for 4 hrs. at 120°C temp. The excess of pyridine was removed by distillation . It was cooled and poured into crused ice and the content was acidfied, with dilute hydrochloric acid to remove the excess of pyridine. The solid obtained was filtered, washed with cold water, dried and recrystallised from ethanol. m.p. 126°C, Yield : 76.48% (Found : C, 56.02; H, 4.50 ; N, 13.06 ; $C_{30}H_{29}O_7N_6F_3$ requires : C, 56.07; H, 4.51; N, 13.08 %) IR (KBr) : 3300 (-NH Str.), 2965 (C-H str.), 1700 (C=O), 1580 (C=N),1485 (-N=O,R-NO_2) 1140 (C-F str.) 1245 (C-O-C asym) ^{1}HNMR (DMSO) :δ 0.85-0.91 (-N-CH_2-CH_2-CH_3), 1.5-1.6 (-N-CH_2-CH_2-CH_3-), 2.9-3.0 (-N-CH_2-CH_2-CH_3-),3.34-3.38(-N-CH_2-CH_2-), 3.64-3.55 (-N-CH_2-CH_2-), 3.90-3.95 (-OCH_3) 7.0-7.9 (Ar-H).

Similarly others compounds (3a-p) were prepared and their physical data are recorded in table - I.

N-N'-(4'-Methoxyphenyl, 4''-methoxyphenyl diamino)phosphinc chloride- (4k). :

A mixture of p-anisidine (0.020 mole), in dry benzene (50 ml) , phosphorusoxychloride (0.01 mole) in dry benzene (50 ml) and triethyl amine (0.5 g) as a basic catalyst in a reaction mixture. The reaction mixture was refluxed for 10 hrs. and cooled, when solid triethylamine hydrochloride seperated out. It was filtered and the filtrate concentracted and dried. recrystallised from dioxane. m.p. 186°C, Yield : 75.86 % (Found : C, 51.49; H, 4.85 ; N, 8.53 ; $C_{14}H_{16}O_3N_2PCl$ requires : C, 51.53; H, 4.90; N, 8.57 %) IR (KBr) : 3375-3300 (-NH Str.), 2965 (C-H str.), 1725 (C-O str., -$COCH_3$), 1580 (-C=N),1245 (-C-O-C asym) ^{1}HNMR (DMSO) : 3.85 (-OCH_3), 6.93-7.86 (Ar-H) 8.83-8.85 (-NH).

Similarly others compounds (4a-u) were prepared and their physical data are recorded in table - I.

N-N'-(4'-Methoxyphenyl, 4''-methoxyphenyl diamino)phosphonic hydrazino ethyl-N-(propyl)-2,6-dinitro-4-(trifluoromethyl)-aniline.- (5k). :

A mixture of compound -(1) (3.51g, 0.01 mole),and triethylamine (0.5 g) in dry benzene (50 ml) was added to a solution of N,N'-(4'-Methoxyphenyl,4''-Methoxyphenyl diamino) phospinic chloride(3.26g, 0.01 mole) in dry benzene (50 ml) . The reaction mixture was refluxed for 10 hrs. and when solid triethylaminehydrochloride seperated out. It was filtered and the filtrate concentrated and dried recrystallised from dioxane. m.p. 160°C, Yield : 76.41% (Found : C, 49.13; H, 4.79 ; N, 15.25 ; $C_{26}H_{31}O_7N_7PF_3$ requires : C, 49.16; H, 4.83; N, 15.29 %) IR (KBr) : 3300-3240 (N-H Str.), 2950 (C-H str.), 1640 (C-O str. -OCH_3), 1370 (-N=O,R-NO_2 str.),1130-1200 cm^{-1} (C-F str.) ^{1}HNMR (DMSO) :δ 0.86-0.91 (-N-CH_2-CH_2-CH_3), 1.54-1.67 (-N-CH_2-CH_2-CH_3) , 2.99-3.04 (-N-CH_2-CH_2-CH_3)3.34-3.36 (-N-CH_2-CH_2-), 3.62-3.67(-N-CH_2-CH_2-), 3.85 (-OCH_3), 7.12-8.17 (Ar-H) , 8.17(-NH).

Similarly others compounds (5a-u) were prepared and their physical data are recorded in table - I.

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