Available online at www.elixirpublishers.com (Elixir International Journal)

Organic Chemistry

Elixir Org. Chem. 94 (2016) 40081-40093

ISSN: 2229-712X

Synthesis of a Novel Series of 2-Substituted Imino-6-Amino-4-[2-Isobutoxy-5(4-Methyl-5-Carboxy-1,3-Thiazo-2-yl)]-Phenyl-1,3,5-Thiadiazines

Tayade D.T. and Shendge A.S.

Department of Chemistry, G.V.I.S.H., Amravati.

ARTICLE INFO

Article history:
Received: 7 January 2016;
Received in revised form:
1 May 2016;
Accepted: 5 May 2016;

Keywords
IR,
NMR,
Mass,
XRD.

ABSTRACT

A novel series of 2-substitutedimino-6-amino-4-[2-isobutoxy-5(4-methyl-5-carboxy-1,3-thiazo-2-yl)]-phenyl-1,3,5-thiadiazineshad been recently synthesized in this laboratory by refluxing 2-(3-substitutedthioamidoforma midino-4-isobutoxyphenyl)-4-methyl-5-carboxy-1,3-thiazoles with various isocyanodichlorides in acetone-ethanol medium in 1:1 molar proportion for 2 hours. The structures of all the synthesized compounds were justified on the basis of chemical characteristics, elemental analysis and spectral studies.

Introduction

Heterocyclic compounds are more intriguing due to their utility in various fields. The literature survey reveals that when the compounds containing 1,3,5-dithiazino or 1,3,5-thiadiazino molecule as a parent nucleus then that molecule will enhance potency of that drug in medicinal, agricultural and industrial fields[1-9].Hence, nowadays the drug containing 1,3,5-dithiazino or 1,3,5-thiadiazino nucleus are widely used in the drug[10-16]. It has been reported that thiadizino nucleus and its derivatives possesses antiviral, antifungal, antibacterial, anti-tuberculostatic and anti-helminthic activities[17-18]. Several thiadiazines are used in the treatment of cancer[19] and some are anti-HIV[20-21] drugs. They are also used in agriculture[22] as fungicidal[23], insecticide[24] while 1,3,5-dithiazines are also effective against copper corrosion[25] and used in lubricating oil[26]. The various researchers have been investigated briefly essential reactions of substituted isocyanodichlo- rides[27-32]. In this laboratory various alternative routes for the synthesis of nitrogen, nitrogen and sulphur containing heteroacycles and heterocycle had been developed. It is quite intriguing to investigate one step cyclisation of 2-(3-substitutedthioamidoformamidino-4-isobutoxyphenyl)-4-methyl-5-carboxy-1,3-thiazoles (IIIa-e) with N-substitutedisocyanodi-chlorides (VIIa-h) in acetone-ethanol medium to isolate 2-substitutedimino-6-amino-4-[2-isobutoxy-5(4-methyl-5-carboxy-1,3-thiazo-2-yl)]-phenyl-1,3,5-thidiazines(VIIIa1-e37).

Materials and Methods

The melting points of all the synthesized compounds were recorded using hot paraffin bath and areuncorrected. The purity of the compounds was checked on Silica Gel-G plates by TLC with layer thickness of 0.3 mm. All chemicals used were of AR grade (Indiamake). Alkyl/arylisothiocyanates, isothiocarbamoylchloride, isocyanodichlo- rides and phenylthiourea have been prepared by known literature method. The carbon and hydrogen analysis was carried out on Carlo-Ebra-1106 analyser. Nitrogen estimations were carried out on Colman-N-analyser-29.

IR spectra were recorded on Perkin-Elmer spectrometer in the range 4000-400 cm^{-1} in KBr pellets.

PMR spectra were recorded on Bruker AC-400F spectrometer with TMS as internal standard using $CDCl_3$ and DMSO-d_6 as solvent.

MASS spectra were recorded on WATERS, Q-TOFmicromass (LC-MS).

During the XRD analysis of the compounds, configuration is equal to reflection spinner stage having owner equal to jagtar. Goniometer is equal to PW3050/60 Theta minimum step size is 0.001 Omega. The diffractometer system which is used during the analysis is of XPERT-PRO. Scan axis is Gonio. Carbon hydrogen and nitrogen analysis and all spectral analysis were carried out at P. U.Chandigarh.

Result and Discussion

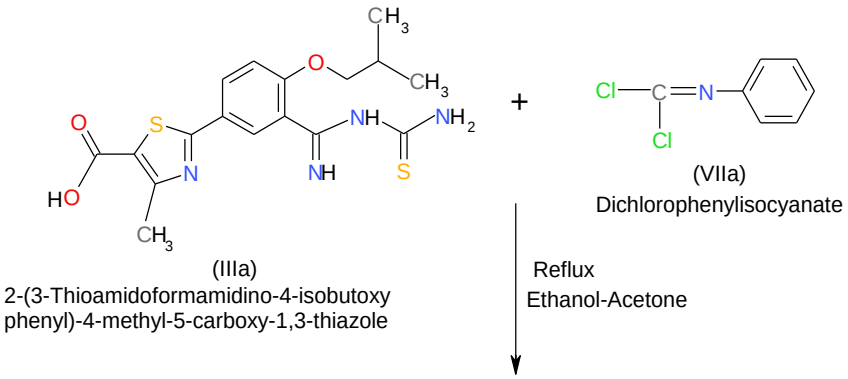
Synthesis of 2-phenylimino-6-amino-4-[2-isobutoxy-5(4-methyl-5-carboxy-1,3-thiazo-2-yl)] -pheny l-1, 3, 5-thiadiazine(VIIIa4)

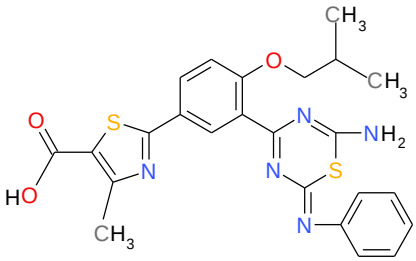
The interaction of 2-(3-thioamido-formamidino-4-isobutoxyphenyl)-4-methyl-5-carboxy-1,3-thiazole (IIIa) with phenyliso-cyanodichloride(VIIa) in 1:1 molar ratio was refluxed on water bath in acetone-ethanol medium for 2 hours. During heating evolution of hydrochloride gas was clearly noticed. After distillation of excess of acetone-ethanol ivory colour product was isolated which on basification with dilute ammonium hydroxide white crystalline products afforded, yield 88%, m.p. 158^{0}C.

Tele:
E-mail address: anilsphd@gmail.com

The probable mechanism for the formation of (VIIIa4) is depicted below,

Reaction





(VIIIa4)
2-Phenylimino-6-amino-4-[-2-isobutoxy-5(-4-methyl-5-carboxy-1,3-thiazo-2-yl)]-phenyl-1,3,5-thiadiazine.

Properties of (VIIIa4)

1. It is white crystalline solid having m.p.158^{0}C.
2. It gave positive test for nitrogen and sulphur.
3. It gave positive test for carboxylic group.
4. It does not desulphurized when boiled withsodiumplumbite solution which is clearly indicates that sulphur is not free and gets cyclised[33-34].
4. It was soluble in benzene, acetic acid, DMF and DMSO.

5. Elemental analysis

The result of elemental analysis is given in Table No. V-1

Table No. V-1

Elements	Found (%)	Calculated (%)
Carbon	57.50	58.41
Hydrogen	04.00	04.66
Nitrogen	14.91	14.91
Sulphur	11.62	12.98

6. From the analytical data the molecular formula was found to be $C_{25}H_{25}N_5O_3S_2$.

7. IR Spectrum

The IR spectrum of compound was carried out in KBr pellets and reproduced on IRPlate No. PPB-14 an important absorption are correlated as follows in Table No.V-2.

Table No. V-2

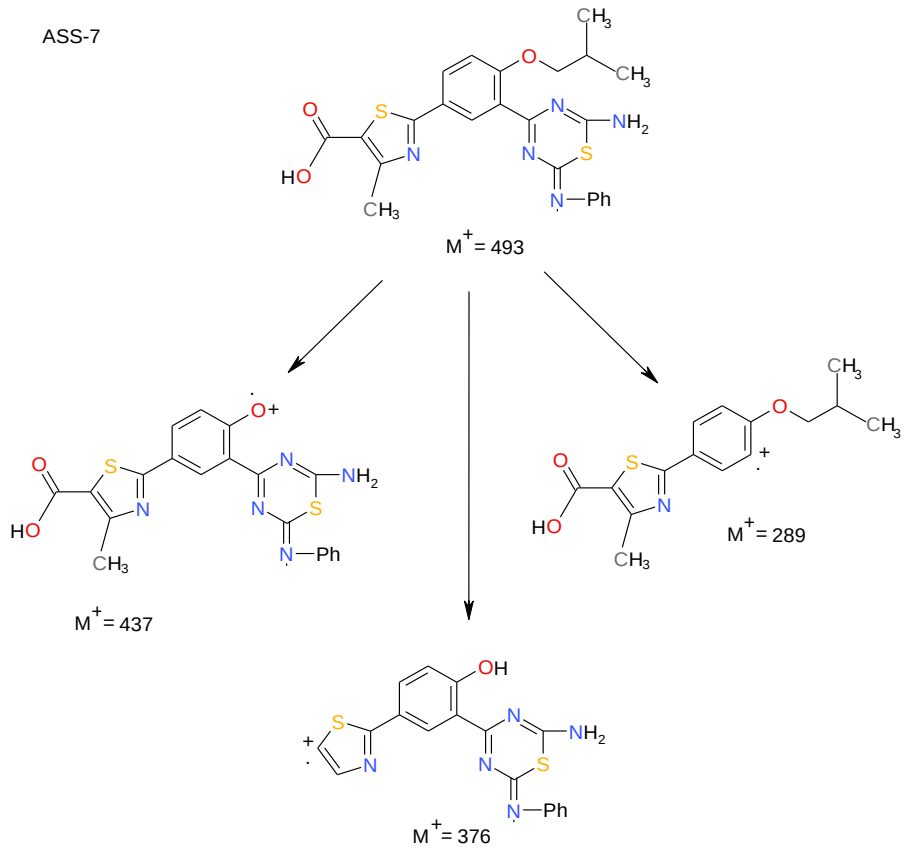
Absorption Observed (cm^{-1})	Assignment[35-37]	Absorption Expected (cm^{-1})
3460.48	-NH Stretching	3500-3000
3105.48	-C-Hstretching	3150-3000
2228.58	-S-C=Nstretching	2270-1940
1706.17	-C=Nstretching	1750-1450
1285.23	-C-N stretching	1360-1000
0738.42	-C-Sstretching	800-600

PMR-Spectrum

1. The PMR spectrum[38] of compoundwas carried out in $CDCl_3$ and DMSO-d_6 and reproduced on PMR Plate No. PPB-14 This spectrum distinctly displayed the signals due -COOH proton at 9.9481 ppm, Ar-H protons at 8.2077-7.9343 ppm, monosubstituted phenyl protons at 7.1516-7.1293 ppm, $-NH_2$ protons at δ 3.6368-3.4143 ppm, $-OCH_2$ protons at δ 2. 5457-2.4074 ppm, -CH proton at δ 1.9079-1.8622 ppm, $-CH_3$ protons at δ 0.9935-0.6244 ppm.

2. **Mass spectrum**

The Mass analysis of compound VIIIa4 was carried out and reproduced on Mass Plate No. ASS-7. The fragmentations occur during the analysis is given in Mass Scheme-IV.



Mass Scheme-IV

3. **XRD Analysis**

The XRD Analysis of compound **VIIIa4** was carried out, during analysis the start position is 02 Th which shows reading from 5.0083 and end position 02 Th is 69.9984. It takes 29.845 second. The analysis of this compound was carried out at 25^0C. Copper is used as anode material. The result and values obtained during XRD are as given below,

<?xml version="1.0" encoding="UTF-8"?>
<xrdMeasurementsxmlns="http://www.xrdml.com/XRDMeasurement/1.3" xmlns:xsi="http://www.w3.org/2001/XMLSchema-instance" xsi:schemaLocation="http://www.xrdml.com/XRDMeasurement/1.3 http://www.xrdml.com/XRDMeasurement/1.3/XRDMeasurement.xsd" status="Completed">
<comment>
<entry>Configuration=Flat Sample Stage, Owner=jagtar, Creation date=6/11/2007 3:57:00 PM</entry>
<entry>Goniometer=PW3050/60 (Theta/Theta); Minimum step size 2Theta:0.001; Minimum step size Omega:0.001</entry>
<entry>Sample stage=PW3071/xx Bracket</entry>
<entry>Diffractometer system=XPERT-PRO</entry>
<entry>Measurement program=PU, Owner=jagtar, Creation date=4/15/2008 1:52:59 PM</entry>
</comment>
<sample type="To be analyzed">
<id></id>
<name></name>
<preparedBy></preparedBy>
</sample>
<xrdMeasurementmeasurementType="Scan" status="Completed" sampleMode="Reflection">
<comment>
<entry/>
</comment>
<usedWavelength intended="K-Alpha 1">
<kAlpha1 unit="Angstrom">1.5405980</kAlpha1>
<kAlpha2 unit="Angstrom">1.5444260</kAlpha2>
<kBeta unit="Angstrom">1.3922500</kBeta>
<ratioKAlpha2KAlpha1>0.5000</ratioKAlpha2KAlpha1>
</usedWavelength>
<incidentBeamPath>
<radius unit="mm">240.00</radius>
<xRayTube id="1010041" name="PW3373/00 Cu LFF DK403331">
<tension unit="kV">45</tension>
<current unit="mA">40</current>

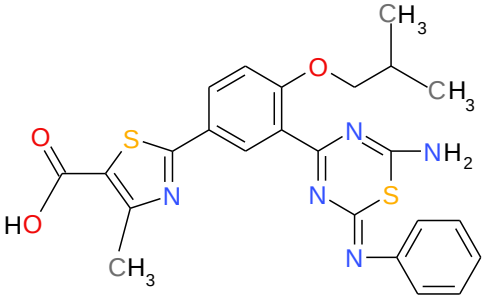
<anodeMaterial>Cu</anodeMaterial>
<focus type="Line">
<length unit="mm">12.0</length>
<width unit="mm">0.4</width>
<takeOffAngle unit="deg">6.0</takeOffAngle>
</focus>
</xRayTube>
<filter id="20010004" name="Nickel">
<material>Ni</material>
<thickness unit="mm">0.020</thickness>
</filter>
<sollerSlit id="21010001" name="Soller 0.02 rad.">
<opening unit="rad">0.0200</opening>
</sollerSlit>
<mask id="22080002" name="Inc. Mask Fixed 10 mm (MPD/MRD)">
<width unit="mm">6.60</width>
</mask>
<antiScatterSlit id="22010003" name="Slit Fixed 1Â°" xsi:type="fixedAntiScatterSlitType">
<height unit="mm">1.52</height>
</antiScatterSlit>
<divergenceSlit id="22010004" name="Slit Fixed 1/2Â°" xsi:type="fixedDivergenceSlitType">
<distanceToSample unit="mm">140.00</distanceToSample>
<height unit="mm">0.76</height>
</divergenceSlit>
</incidentBeamPath>
<diffractedBeamPath>
<radius unit="mm">240.00</radius>
<antiScatterSlit id="22090004" name="AS Slit 5.5 mm (X'Celerator)" xsi:type="fixedAntiScatterSlitType">
<height unit="mm">5.50</height>
</antiScatterSlit>
<sollerSlit id="21010001" name="Soller 0.02 rad.">
<opening unit="rad">0.0200</opening>
</sollerSlit>
<detector id="7010013" name="X'Celerator" xsi:type="rtmsDetectorType">
<phd>
<lowerLevel unit="%">40.5</lowerLevel>
<upperLevel unit="%">79.0</upperLevel>
</phd>
<mode>Scanning</mode>
<activeLength unit="deg">2.122</activeLength>
</detector>
</diffractedBeamPath>
<scan appendNumber="0" mode="Continuous" scanAxis="Gonio" status="Completed">
<startTimeStamp>2015-07-30T15:13:27+05:30</startTimeStamp>
<endTimeStamp>2015-07-30T15:29:16+05:30</endTimeStamp>
<author>
<name>Panjab University</name>
</author>
<source>
<applicationSoftware version="2.2j">X'Pert Data Collector</applicationSoftware>
<instrumentControlSoftware version="2.1A">XPERT-PRO</instrumentControlSoftware>
<instrumentID>0000000011023505</instrumentID>
</source>
</header>
<dataPoints>
<positions axis="2Theta" unit="deg">
<startPosition>5.00835563</startPosition>
<endPosition>69.99848069</endPosition>
</positions>
<positions axis="Omega" unit="deg">
<startPosition>2.50517782</startPosition>
<endPosition>35.00024035</endPosition>
</positions>
<commonCountingTime unit="seconds">29.845</commonCountingTime>

<intensities unit="counts">544 529 565 582 510 513 525 515 589 527 601 569 579 543 579 515 574 572 551 583 550 537 509
517 530 545 502 508 592 544 531 561 540 577 550 520 555 544 579 571 573 520 532 507 559 538 555 570 536 534 545 506 531
520 542 524 505 508 556 533 523 537 525 521 532 522 529 493 517 528 544 508 487 555 535 527 535 525 515 506 563 559 507
518 493 532 504 523 510 509 476 507 555 504 557 503 523 573 507 534 483 490 482 495 514 511 540 551 502 518 538 529 542
547 478 523 515 499 476 523 522 504 533 518 516 508 566 501 502 543 497 504 486 500 534 515 507 508 496 534 549 487 501
476 471 492 542 554 532 496 515 543 531 520 543 490 508 520 544 492 483 517 477 513 527 542 555 528 493 494 518 517 542
497 492 524 552 471 488 538 500 536 499 509 525 542 500 495 542 478 548 540 500 548 507 531 537 524 527 537 520 521 531
525 551 510 530 505 544 522 501 545 495 549 519 521 496 536 516 540 551 507 519 503 485 537 497 537 478 500 524 541 480
515 511 496 497 565 547 504 520 502 509 541 594 516 583 511 592 580 566 538 547 506 506 529 547 535 560 541 549 545 550
558 610 588 621 505 556 576 586 614 615 613 605 618 624 604 580 586 601 572 564 555 560 503 534 572 518 564 575 536 554
536 541 563 557 583 569 580 621 589 525 536 599 594 565 608 536 535 574 582 531 548 562 534 568 620 562 573 556 591 589
520 565 607 497 559 571 574 527 555 605 563 576 555 529 586 568 550 584 570 551 541 530 544 558 566 574 594 529 574 589
600 559 547 575 604 577 573 597 579 575 517 590 607 560 575 616 615 592 580 622 597 574 588 578 633 574 598 588 578 565
665 628 594 603 549 642 612 648 598 611 585 617 641 611 569 628 586 638 592 578 577 570 624 551 585 620 627 617 643 611
631 620 626 580 604 643 569 548 600 620 618 638 577 569 597 611 599 613 618 573 625 570 602 592 572 611 654 629 637 572
572 660 602 585 610 598 606 579 627 544 613 616 665 602 599 660 612 645 582 572 626 638 658 639 657 638 632 677 585 633
596 596 674 668 612 616 628 615 629 612 655 652 641 676 695 660 682 662 641 667 626 686 669 663 689 631 662 641 668 694
668 661 661 675 646 654 679 724 641 649 716 658 686 731 686 743 698 756 722 753 711 708 667 702 730 713 716 738 668 711
765 710 703 792 742 791 755 728 762 728 744 680 799 782 765 796 745 759 824 821 772 752 790 902 815 842 823 813 821 848
868 784 848 890 922 860 909 853 863 901 888 933 944 930 947 992 930 929 924 953 1007 942 1028 971 937 1003 1043 1009
1025 1064 1054 1012 1049 1051 1071 1026 1131 1044 1139 1081 1048 1144 1132 1186 1074 1130 1116 1171 1059 1116 1100
1073 1143 1183 1088 1108 1140 1074 1097 1097 1000 1080 1068 1171 1047 1019 1032 1118 1013 1031 1071 1081 1031 985
1050 1057 999 977 1006 1001 1033 1035 994 1011 920 987 991 953 1016 954 985 1030 1036 1004 953 994 940 976 911 970
949 1021 928 1000 966 938 938 1003 972 1003 933 993 963 999 1043 980 945 1045 989 1020 975 992 997 992 1006 1036 1009
1028 1017 1048 1033 1109 1068 1085 1042 1075 1037 1047 1039 1009 1105 1091 1139 1105 1097 1098 1136 1058 1084 1060
1086 1128 1182 1090 1148 1116 1064 1103 1119 1033 1155 1131 1085 1175 1091 1063 1128 1118 1068 1068 1098 1072 1086
1188 1108 1096 1110 1132 1070 1086 1035 1043 1040 1016 1029 1035 997 1070 1088 1023 977 1053 975 1014 1016 1049 1086
998 980 1034 1076 1055 1048 991 1033 992 1014 1027 1009 1090 1087 1063 977 1044 1078 1044 1059 1077 1015 1025 1046
1097 1061 1025 1055 1037 1036 1097 1062 1098 1002 980 1098 1013 1036 1008 1009 973 998 1002 1014 1047 1102 1101 1118
1034 988 1002 977 921 876 918 970 915 882 878 862 910 900 859 873 857 873 881 840 854 838 838 866 826 853 862 845 790
922 865 826 798 826 832 853 790 847 874 853 822 840 848 812 867 825 832 819 829 860 842 853 852 857 888 853 842 819 850
922 908 850 856 860 836 827 923 837 818 864 811 852 898 854 885 919 840 852 882 902 880 870 904 869 943 968 912 910 847
905 877 891 936 930 915 925 943 907 936 878 904 895 926 902 938 943 904 942 895 911 951 923 892 933 949 962 938 911 943
946 940 918 910 1003 942 867 936 871 985 914 829 893 967 931 937 939 938 966 887 917 956 879 917 924 912 870 912 936
935 888 856 956 971 916 898 933 934 895 908 879 950 893 880 982 922 954 956 893 920 909 918 947 979 946 935 919 994
1034 985 981 985 1011 974 991 1065 1043 1018 1026 1029 986 1047 1084 1028 1049 1059 1082 1028 1171 1078 1096 1136
1148 1154 1158 1223 1220 1157 1194 1221 1230 1252 1192 1217 1249 1291 1285 1382 1296 1385 1290 1317 1357 1372 1333
1358 1420 1379 1383 1455 1490 1484 1560 1499 1513 1563 1570 1601 1591 1609 1586 1565 1620 1572 1616 1723 1666 1655
1734 1711 1759 1680 1667 1723 1742 1759 1767 1629 1781 1682 1712 1773 1740 1690 1681 1693 1710 1777 1707 1690 1643
1663 1608 1618 1625 1681 1656 1620 1573 1497 1525 1500 1534 1529 1435 1512 1487 1372 1506 1468 1406 1425 1379 1280
1382 1309 1334 1303 1208 1223 1225 1271 1197 1256 1120 1148 1123 1113 1134 1105 1142 1162 1050 1015 1111 1072 1094
1029 1036 1082 1022 1005 1023 1007 954 1009 954 951 908 1002 1058 967 942 960 921 961 902 942 953 919 948 896 854 878
800 907 811 847 808 848 870 807 809 769 808 859 817 779 787 782 791 843 808 767 836 771 777 804 750 776 739 778 731 746
808 790 767 785 773 764 767 697 701 687 738 688 690 712 730 697 726 725 710 663 705 683 707 666 699 737 687 704 702 669
699 717 652 727 682 640 645 683 655 672 668 698 620 718 656 640 610 627 618 659 599 655 647 646 648 611 599 651 641 659
628 610 620 592 627 613 600 627 611 688 577 652 577 590 681 606 611 582 608 600 613 642 557 611 586 613 591 572 569 610
644 594 563 631 572 651 656 605 596 635 605 589 610 554 663 621 631 587 601 585 586 587 599 606 593 640 620 632 612 621
558 610 586 573 541 612 570 621 546 560 593 586 557 594 564 551 541 554 573 576 579 591 577 600 537 531 608 552 616 585
529 554 507 520 537 559 582 533 540 582 572 543 547 532 525 545 523 552 565 546 536 558 585 603 519 529 549 573 552 560
552 521 533 563 568 523 545 517 521 524 569 543 560 634 540 551 588 545 541 546 572 517 587 543 523 503 555 501 519 561
483 555 537 523 524 513 514 517 578 548 546 552 515 525 554 576 508 604 562 545 576 599 584 594 560 576 559 594 610 550
589 597 580 576 592 628 589 565 565 510 574 513 558 558 585 572 523 592 607 611 561 517 565 565 565 573 591 559 594 554
569 531 555 574 555 546 577 545 556 564 537 542 558 560 564 565 576 579 573 576 569 611 614 555 578 586 549 548 588 519
525 527 551 545 531 564 525 524 531 530 552 590 561 542 570 507 521 551 550 578 560 545 573 600 568 554 588 654 568 626
575 597 601 569 607 573 552 574 558 603 513 573 594 521 544 541 550 555 576 574 572 559 597 564 563 548 534 585 520 522
548 542 591 596 535 513 531 513 579 515 528 514 524 538 537 530 525 509 518 524 564 551 524 577 531 491 523 508 555 558
570 560 508 515 473 550 502 549 543 550 502 502 530 510 523 559 511 505 546 466 535 495 540 573 467 491 521 538 524 546
461 489 516 486 478 487 524 518 472 505 535 515 518 503 442 464 487 478 535 471 471 481 497 496 556 533 485 448 475 492
444 524 455 483 489 466 487 518 478 480 518 479 508 518 507 523 508 501 524 499 472 469 481 486 505 556 493 492 496 494
481 469 485 474 505 501 523 475 537 543 459 511 528 457 525 487 486 536 485 500 459 522 502 466 490 499 489 492 499 497
529 523 495 534 521 568 492 524 482 495 505 519 511 519 524 507 547 532 465 534 544 544 534 524 546 496 521 514 468 570
564 520 573 545 533 519 578 554 624 601 569 651 618 594 563 590 575 588 588 596 558 565 532 577 570 570 515 541 516 516
551 560 599 529 535 614 564 542 544 522 555 546 571 584 528 587 557 549 608 619 550 597 614 617 613 606 597 579 597 603
579 541 556 607 578 558 557 546 578 541 545 527 555 533 507 490 523 518 501 540 517 474 488 514 489 483 500 514 524 478

529 476 494 505 465 518 506 466 494 525 496 503 471 490 520 448 474 488 452 503 464 507 510 486 471 501 435 475 468 461
504 519 495 492 465 455 513 458 463 489 449 481 440 499 493 515 528 474 502 482 465 474 477 528 473 488 474 441 477 518
469 466 472 488 465 474 533 466 496 461 450 465 479 493 463 485 459 451 442 494 423 468 480 493 473 423 489 469 486 417
473 457 469 439 463 450 471 458 446 438 472 441 425 421 399 408 442 434 450 483 455 445 430 445 442 425 433 399 427 452
437 473 450 389 438 433 400 428 405 446 431 407 434 419 430 399 400 437 392 391 428 408 411 444 385 445 434 431 402 406
382 409 402 441 450 372 430 418 405 398 442 412 392 467 430 394 408 406 454 430 408 452 413 409 404 445 457 411 414 404
390 467 403 431 411 369 439 450 402 425 392 421 396 440 435 409 397 406 395 355 441 468 446 407 398 424 395 395 407 442
422 430 448 416 413 404 434 419 408 411 407 424 396 406 433 404 426 416 390 432 438 405 435 433 405 399 392 434 444 394
389 427 374 388 418 414 411 428 411 438 393 380 409 451 410 369 396 417 422 422 372 380 410 418 377 402 412 421 436 389
361 355 380 375 393 389 386 379 402 365 386 347 389 371 375 399 380 382 405 386 365 388 382 372 381 381 338 361 372 356
402 408 384 317 366 366 409 374 394 347 351 333 371 379 364 378 374 382 370 360 381 386 353 406 345 365 383 390 362 365
345 360 357 349 375 360 334 343 425 344 361 331 390 382 358 342 383 371 360 348 384 377 414 357 369 369 356 369 337 362
366 380 324 361 384 327 378 396 328 383 382 376 352 368 379 375 400 366 365 326 323 393 339 363 365 344 350 353 312 307
361 377 363 388 342 328 358 358 352 341 349 383 338 349 358 367 374 377 360 316 341 336 355 312 347 354 394 397 322 356
356 343 354 349 336 329 371 379 371 351 340 374 355 366 340 313 302 319 343 366 349 376 394 335 343 383 363 374 369 357
355 345 348 351 331 363 347 345 392 325 355 338 378 354 388 326 328 320 340 323 336 354 370 354 323 339 365 341 335 320
328 348 358 329 322 348 325 337 308 349 316 328 327 328 349 301 379 356 342 357 342 336 315 306 324 362 345 328 315 334
338 354 362 365 323 343 314 317 356 333 267 294 308 316 315 323 324 300 310 371 343 330 342 315 303 324 349 344 326 352
312 351 339 365 328 323 350 308 305 317 344 322 316 291 351 329 308 324 338 347 376 347 329 307 345 331 345 325 312 360
331 340 311 287 326 311 341 348 329 330 368 347 331 337 341 330 348 358 342 369 341 337 341 322 334 336 314 306 320 313
323 284 335 313 351 335 314 320 342 305 309 317 333 324 322 296 341 340 313 327 354 330 350 310 322 303 330 305 323 310
310 312 294 325 315 306 296 269 306 336 334 352 311 307 328 324 305 321 299 287 285 340 333 310 364 292 311 310 309 334
349 316 344 284 315 305 329 297 295 314 334 300 295 337 307 298 298 326 284 281 294 283 298 306 319 350 315 355 316 333
309 305 300 299 291 310 293 321 312 300 355 330 320 303 314 308 273 340 327 326 305 301 310 313 307 305 277 317 300 330
307 344 337 297 364 306 291 343 283 361 303 336 300 300 317 334 323 305 315 313 291 299 314 301 315 348 343 277 300 328
324 295 303 307 295 315 268 302 333 297 317 302 300 288 316 321 317 305 297 328 298 324 298 342 308 330 280 280 303 301
278 282 301 307 266 315 316 294 313 301 289 282 307 339 283 291 333 286 305 299 281 308 311 321 313 331 356 354 322 335
326 301 283 284 288 288 306 283 284 277 297 320 291 265 258 317 241 248 261 283 264 242 261 300 282 317 274 301 283 273
285 270 256 256 246 252 274 257 287 240 293 272 271 259 274 260 264 240 257 300 267 230 265 285 251 268 255 268 256 273
283 270 247 275 272 267 261 268 271 278 238 270 259 253 281 279 264 240 294 269 255 275 234 252 264 250 261 233 231 229
234 251 244 227 246 255 236 264 265 278 245 239 269 257 258 225 233 232 231 257 239 210 264 254 248 222 256 233 252 236
247 252 259 259 261 232 244 240 255 239 271 237 252 254 229 250 230 269 226 247 254 248 213 229 259 252 251 256 251 241
260 244 236 225 277 243 257 237 268 268 228 257 257 231 243 253 229 217 240 237 247 213 225 248 236 228 237 241 214 233
242 209 224 246 246 265 239 240 248 224 236 236 237 223 257 245 245 250 230 195 228 224 236 219 256 227 216 233 196 230
233 199 238 199 217 215 232 224 233 219 217 240 255 230 230 202 214 219 226 224 208 216 222 225 239 209 218 245 224 204
220 189 208 203 248 278 196 203 231 230 211 211 230 209 258 206 231 231 223 224 198 185 231 197 219 212 229 233 228 212
210 226 218 214 250 236 204 203 212 246 209 199 213 190 234 209 217 205 246 237 190 252 200 201 234 204 210 202 241 213
231 215 209 257 205 216 234 224 228 205 201 248 217 205 233 209 213 211 208 228 185 244 212 223 200 218 203 218 192 248
177 232 234 209 192 224 225 234 216 217 227 182 220 248 191 201 186 219 209 210 215 216 215 216 211 202 199 196 237 225
225 198 198 210 196 214 209 202 211 204 203 198 218 178 190 198 197 205 181 203 220 199 205 201 185 198 208 216 192 196
204 210 187 177 204 192 194 200 197 184 204 217 202 214 227 184 197 216 222 212 209 201 195 213 218 205 222 183 176 192
215 201 186 202 197 211 185 208 206 205 186 203 198 196 184 201 202 189 191 168 196 206 180 188 197 194 188 199 200 190
182 198 175 167 202 184 195 214 202 189 205 173 201 198 196 198 201 184 167 189 173 204 191 206 205 171 176 172 189 189
184 190 180 205 197 196 190 184 219 210 174 180 182 173 178 204 175 185 192 187 198 178 200 186 173 201 168 177 162 177
194 196 189 189 182 199 177 192 187 156 198 187 171 191 187 188 187 167 184 185 172 163 190 192 192 201 199 178 197 201
186 206 185 209 184 176 194 178 169 169 186 209 165 165 178 196 163 184 198 173 197 190 200 173 198 157 190 183 176 191
171 225 195 158 204 177 172 184 161 187 181 184 169 181 177 171 206 177 167 163 175 173 183 182 177 208 169 177 172 188
183 162 187 187 175 172 162 186 165 187 181 188 159 185 158 177 181 187 189 157 170 169 192 163 173 172 175 169 195 170
169 177 169 178 167 174 172 177 160 173 183 168 163 158 149 169 190 171 165 181 177 159 167 137 189 177 168 170 156 199
196 168 176 177 153 190 158 172 170 190 162 176 198 178 171 171 158 189 194 164 169 175 195 163 187 160 180 161 169 169
181 176 171 173 150 158 183 169 187 185 145 172 184 177 162 170 192 178 180 182 183 201 196 208 165 177 168 194 164 174
191 184 175 172 151 185 184 167 172 187 192 187 156 198 204 165 159 145 170 148 184 175 183 184 188 188 172 172 174 171
160 187 178 189 158 167 146 157 168 156 155 174 177 165 176 182 146 142 173 154 144 162 192 167 178 163 163 195 161 176
170 153 155 154 175 177 167 158 121 163 173 175 171 167 155 156 135 155 177 147 175 174 164 164 178 151 151 149 155 161
171 173 174 188 146 155 165 160 156 169 162 141 155 172 157 137 171 144 158 172 149 155 169 153 196 145 162 151 158 168
159 169 148 141 147 156 185 161 157 151 162 178 153 165 162 157 144 162 142 149 158 165 159 149 149 161 164 164 187 140
158 114 181 160 143 190 149 155 148 159 165 142 144 146 169 149 169 186 142 175 160 148 157 154 137 131 180 122 142 134
156 176 151 178 161 147 141 157 168 132 130 150 153 146 140 158 162 170 158 149 163 158 132 154 148 162 147 158 164 146
159 148 176 147 153 154 159 146 149 132 135 153 158 151 168 154 137 162 137 153 146 157 156 158 142 150 160 164 147 184
155 151 167 167 147 150 137 154 144 151 177 167 159 174 174 168 168 183 140 143 154 135 140 148 140 150 167 161 119 154
150 140 182 145 155 163 153 164 157 162 151 148 147 159 136 147 182 159 177 149 147 159 167 170 162 150 151 145 167 143
153 156 165 174 153 164 161 158 173 151 158 148 137 150 128 157 160 158 140 144 175 146 161 147 167 151 156 155 151 136
155 140 145 156 169 151 159 146 147 143 147 136 136 146 156 158 148 153 153 128 161 155 162 134 164 158 157 151 136 148
167 167 149 139 160 158 162 161 175 129 165 159 158 164 141 159 151 162 135 161 149 160 150 169 143 146 157 153 153 125

157 167 146 139 158 174 150 144 171 145 148 156 139 144 151 172 157 139 161 153 150 139 134 162 159 140 141 145 155 136 151 156 147 136 160 146 165 164 146 130 157 134 142 157 151 125 127 141 169 146 144 147 186 157 118 163 134 142 157 145 146 122 142 132 141 146 150 143 118 149 154 147 143 133 168 131 134 141 134 136 146 141 162 144 139 168 135 180 157 137 114 146 157 125 121 148 137 150 144 141 128 145 137 133 127 136 141 158 139 133 141 170 132 139 136 160 145 144 159 144 129 134 148 154 158 144 144 137 134 141 134 135 180 143 141 141 134 149 142 111 134 143 147 135 148 130 140 144 141 129 148 141 147 132 153 146 142 151 149</intensities>

From the above properties and spectral analysis of the compound (VIIIa4) was assigned the structure as 2-phenylimino-6-amino-4-[2-isobutoxy-5(4-methyl-5-carbo -xy-1,3-thiazo-2-yl)]-phenyl-1,3,5-thiadi- azine(VIIIa4)



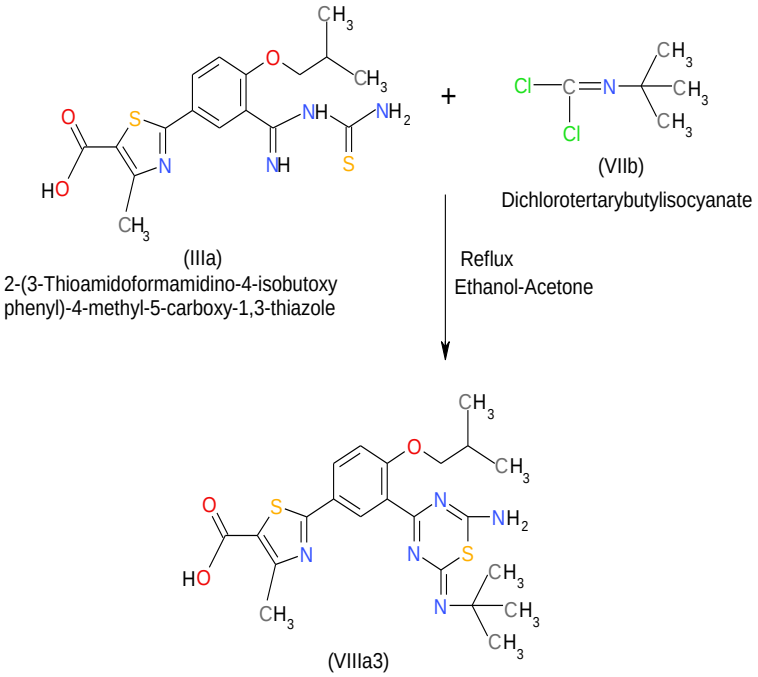
(VIIIa4)

2-Phenylimino-6-amino-4-[-2-isobutoxy-5(-4-methyl-5-carboxy-1,3-thiazo-2-yl)]-phenyl-1,3,5-thiadiazine.

Synthesis of 2-tert-butylimino-6-amino-4-[2-isobutoxy-5(4-methyl-5-carboxy-1,3-thiazo-2-yl)]-phenyl-1,3,5-thiadiazine(VIIIa3)

The interaction of 2-(3-thioamidoformamidino-4-isobutoxyphenyl)-4-meth -yl-5-carboxy-1,3-thiazole (IIIa) with tert-butylisocyanodichloride(VIIb) in 1:1 molar ratio was refluxed on water bath in acetone-ethanol medium for 2 hours. During heating evolution of hydrochloride gas was clearly noticed. After distillation of excess of acetone-ethanol red coloured product was isolated which on basification with dilute ammonium hydroxide brown crystals were afforded, yield 84%, m.p.166^{0}C.

Reaction



(VIIIa3)

2-Tert-butylimino-6-amino-4-[-2-isobutoxy-5(-4-methyl-5-carboxy-1,3-thiazo-2-yl)]-phenyl-1,3,5-thiadiazine.

Properties of (VIIIa3)

1. It is white crystalline solid having m.p.166^{0}C.
2. It gave positive test for nitrogen and sulphur.
3. It gave positive test for carboxylic group.
4. It does not desulphurized when boiled with sodium plumbite solution which clearly indicates that sulphur is not free and gets cyclised[157, 230].
5. It was soluble in benzene, acetic acid, DMF and DMSO.
6. Elemental analysis

The result of elemental analysis is given in Table No. V-3

Table No. V-3

Elements	Found (%)	Calculated (%)
Carbon	54.65	55.81
Hydrogen	04.90	05.70
Nitrogen	14.79	14.79
Sulphur	12.10	13.53

7. From the analytical data the molecular formula was found to be $C_{23}H_{29}N_5O_3S_2$.

8. IR Spectrum

The IR spectrum of compound was carried out in KBr-pellets and reproduced on IRPlate No. PPB-15, an important absorption are correlated as follows in Table No. V-4

Table No. V-4

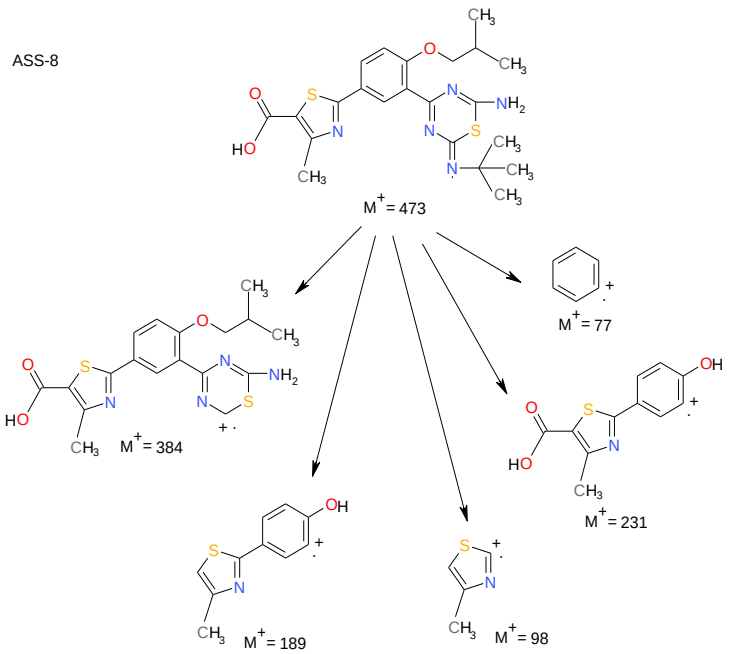
Absorption observed (cm^{-1})	Assignment[231,259-260]	Absorption Expected (cm^{-1})
3452.25	-NH Stretching	3500-3000
3000.00	(Ar)C-Hstretching	3150-3000
1646.47	-C=Nstretching	1750-1450
1634.00	(Ar)C=Cstretching	1600-1450
1107.17	-C-N stretching	1360-1000
0701.48	-C-Sstretching	0800-0600

9. PMR-Spectrum

The PMR spectrum[261] of compoundwas carried out in $CDCl_3$ and DMSO-d_6 and reproduced on PMR Plate No. PPB-15. This spectrum distinctly displayed the signals due to -COOH proton at δ 8.32 ppm,Ar-H protons at δ 6.5049-6.4889 ppm, $-NH_2$ protons at δ 4.9238-4.0207 ppm, $-CH_3$ protons at δ 1.1894-1.2322 ppm, $-O-CH_2$ protons at δ 3.0088-3.8509 ppm, -CH proton at δ 0.8599-0.8432 ppm.

10. Mass spectrum

The Mass analysis of compound VIIIa3 was carried out and reproduced on Mass Plate No. ASS-8. The fragmentations occur during the analysis is given in Mass Scheme-IV



Mass Scheme-IV

11. XRD Analysis

The XRD Analysis of compound VIIIa3 was carried out, during analysis the start position is 02 Th which shows reading from 5.0083 and end position 02 Th is 69.9984. It takes 29.845 second. The analysis of this compound was carried out at 25^{0}C. Copper is used as anode material. The result and values obtained during XRD are as given below,

<xrdMeasurements xmlns="http://www.xrdml.com/XRDMeasurement/1.3"
xmlns:xsi="http://www.w3.org/2001/XMLSchema-instance"
xsi:schemaLocation="http://www.xrdml.com/XRDMeasurement/1.3
http://www.xrdml.com/XRDMeasurement/1.3/XRDMeasurement.xsd" status="Completed">
<comment>
<entry>Configuration=Flat Sample Stage, Owner=jagtar, Creation date=6/11/2007 3:57:00 PM</entry>
<entry>Goniometer=PW3050/60 (Theta/Theta); Minimum step size 2Theta:0.001; Minimum step size Omega:0.001</entry>
<entry>Sample stage=PW3071/xx Bracket</entry>
<entry>Diffractometer system=XPERT-PRO</entry>
<entry>Measurement program=PU, Owner=jagtar, Creation date=4/15/2008 1:52:59 PM</entry>
</comment>
<sample type="To be analyzed">

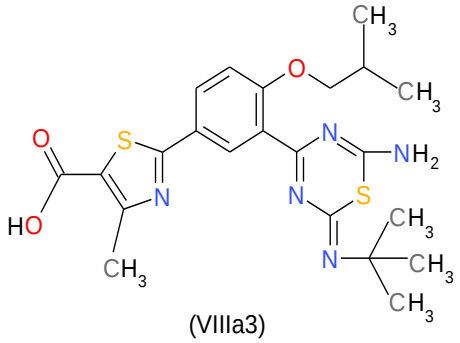
<id></id>
<name></name>
<preparedBy></preparedBy>
</sample>
<xrdMeasurementmeasurementType="Scan" status="Completed" sampleMode="Reflection">
<comment>
<entry/>
</comment>
<usedWavelength intended="K-Alpha 1">
<kAlpha1 unit="Angstrom">1.5405980</kAlpha1>
<kAlpha2 unit="Angstrom">1.5444260</kAlpha2>
<kBeta unit="Angstrom">1.3922500</kBeta>
<ratioKAlpha2KAlpha1>0.5000</ratioKAlpha2KAlpha1>
</usedWavelength>
<incidentBeamPath>
<radius unit="mm">240.00</radius>
<xRayTube id="1010041" name="PW3373/00 Cu LFF DK403331">
<tension unit="kV">45</tension>
<current unit="mA">40</current>
<anodeMaterial>Cu</anodeMaterial>
<focus type="Line">
<length unit="mm">12.0</length>
<width unit="mm">0.4</width>
<takeOffAngle unit="deg">6.0</takeOffAngle>
</focus>
</xRayTube>
<filter id="20010004" name="Nickel">
<material>Ni</material>
<thickness unit="mm">0.020</thickness>
</filter>
<sollerSlit id="21010001" name="Soller 0.02 rad.">
<opening unit="rad">0.0200</opening>
</sollerSlit>
<mask id="22080002" name="Inc. Mask Fixed 10 mm (MPD/MRD)">
<width unit="mm">6.60</width>
</mask>
<antiScatterSlit id="22010003" name="Slit Fixed 1Â°" xsi:type="fixedAntiScatterSlitType">
<height unit="mm">1.52</height>
</antiScatterSlit>
<divergenceSlit id="22010004" name="Slit Fixed 1/2Â°" xsi:type="fixedDivergenceSlitType">
<distanceToSample unit="mm">140.00</distanceToSample>
<height unit="mm">0.76</height>
</divergenceSlit>
</incidentBeamPath>
<diffractedBeamPath>
<radius unit="mm">240.00</radius>
<antiScatterSlit id="22090004" name="AS Slit 5.5 mm (X'Celerator)" xsi:type="fixedAntiScatterSlitType">
<height unit="mm">5.50</height>
</antiScatterSlit>
<sollerSlit id="21010001" name="Soller 0.02 rad.">
<opening unit="rad">0.0200</opening>
</sollerSlit>
<detector id="7010013" name="X'Celerator" xsi:type="rtmsDetectorType">
<phd>
<lowerLevel unit="%">40.5</lowerLevel>
<upperLevel unit="%">79.0</upperLevel>
</phd>
<mode>Scanning</mode>
<activeLength unit="deg">2.122</activeLength>
</detector>
</diffractedBeamPath>
<scan appendNumber="0" mode="Continuous" scanAxis="Gonio" status="Completed">
<header>
<startTimeStamp>2015-07-31T14:42:17+05:30</startTimeStamp>

<endTimeStamp>2015-07-31T14:58:09+05:30</endTimeStamp>
<author>
<name>Panjab University</name>
</author>
<source>
<applicationSoftware version="2.2j">X'Pert Data Collector</applicationSoftware>
<instrumentControlSoftware version="2.1A">XPERT-PRO</instrumentControlSoftware>
<instrumentID>0000000011023505</instrumentID>
</source>
</header>
<dataPoints>
<positions axis="2Theta" unit="deg">
<startPosition>5.00835563</startPosition>
<endPosition>69.99848069</endPosition>
</positions>
<positions axis="Omega" unit="deg">
<startPosition>2.50517782</startPosition>
<endPosition>35.00024035</endPosition>
</positions>
<commonCountingTime unit="seconds">29.845</commonCountingTime>
<intensities unit="counts">421 414 399 391 450 427 426 438 452 451 412 453 420 493 464 433 480 468 451 435 473 439 483
424 420 380 436 386 380 385 386 379 389 389 364 341 374 379 391 368 354 370 356 337 385 376 348 385 357 385 353 375 385
329 369 350 375 371 374 362 370 377 365 374 347 358 362 348 384 347 361 353 338 360 344 364 312 375 377 367 338 353 383
379 313 358 306 378 379 374 315 357 347 352 316 315 344 364 399 354 362 361 328 343 338 388 326 365 367 377 362 351 382
371 354 327 344 399 343 366 353 370 366 368 371 383 345 327 393 412 362 375 382 426 393 421 416 375 390 409 430 378 374
389 370 372 377 362 364 358 371 394 382 405 364 349 367 377 353 386 380 394 417 382 355 414 379 378 365 362 339 375 345
351 320 327 350 326 365 363 324 335 333 317 335 323 291 375 325 330 330 336 308 338 334 327 354 312 299 339 309 341 317
350 302 321 314 322 337 342 322 310 306 327 305 341 358 326 327 354 323 348 308 300 280 340 306 313 336 330 313 311 321
342 320 313 323 313 323 344 300 291 330 306 336 303 327 326 298 293 315 311 310 331 303 293 317 321 296 330 309 313 302
322 286 295 297 289 316 302 288 321 311 336 323 320 297 311 275 335 360 302 293 323 314 256 325 316 313 296 320 343 321
322 330 331 311 286 313 319 282 324 337 317 322 305 299 282 302 293 314 325 307 315 302 323 295 306 310 298 297 287 291
308 315 316 312 303 308 279 294 316 293 262 264 283 258 295 307 272 296 312 285 287 296 260 250 291 278 280 300 330 268
296 299 294 312 308 302 338 295 299 298 274 323 300 270 301 306 292 292 297 268 316 309 289 291 284 254 284 274 242 299
308 273 281 274 273 275 281 268 278 307 287 294 279 287 277 279 309 279 286 296 287 333 319 295 291 306 322 345 306 317
328 344 314 353 337 358 381 352 376 355 324 360 358 377 388 385 393 385 389 367 419 391 424 388 400 397 356 434 386 394
351 333 360 357 317 340 328 350 308 326 337 356 340 342 331 334 333 305 342 306 314 330 311 306 311 306 320 321 325 335
356 324 368 350 371 336 355 344 324 336 364 345 307 326 316 296 316 340 331 313 336 349 314 331 335 336 340 343 351 321
317 342 345 365 301 297 342 296 261 283 319 300 309 297 319 316 300 297 299 291 330 300 301 286 255 273 300 272 296 273
307 303 256 315 265 289 267 293 292 260 325 286 315 305 284 327 280 303 279 306 275 292 299 313 324 296 295 307 288 312
280 303 294 329 315 333 300 288 282 278 330 297 321 294 317 275 310 294 269 317 292 292 286 322 285 316 345 296 311 313
341 309 309 316 284 315 279 303 323 309 317 326 337 312 303 321 317 306 319 327 329 293 325 340 292 278 328 335 308 330
305 300 361 300 300 331 322 272 303 301 348 306 322 328 297 303 347 329 322 319 320 335 334 326 322 327 313 323 339 291
321 286 319 336 325 314 342 309 329 327 330 298 356 343 308 322 372 336 368 335 305 354 369 340 314 339 379 338 370 361
371 363 368 405 379 428 436 436 370 425 450 424 427 409 395 375 390 381 405 412 368 379 410 416 403 372 395 410 365 386
403 390 356 349 381 366 323 288 362 333 321 379 357 360 349 339 345 356 357 350 341 305 349 361 320 328 320 335 338 344
344 356 307 326 278 311 333 292 307 321 363 311 333 294 296 343 323 285 305 314 310 286 305 348 361 314 310 341 339 305
351 314 316 344 337 312 336 348 314 338 297 295 292 324 324 343 350 323 300 320 291 280 299 303 316 288 311 287 300 330
284 259 293 275 282 312 303 298 271 335 275 272 279 319 275 314 281 305 289 339 274 302 312 313 312 283 335 279 285 321
289 323 302 283 312 305 308 317 289 333 368 306 324 309 342 347 331 372 371 375 339 384 361 363 349 369 378 398 411 390
417 437 402 353 398 399 426 416 411 531 643 722 759 704 621 521 474 448 366 399 431 390 360 389 360 421 417 492 413 450
452 446 500 441 465 483 464 504 485 497 479 460 502 503 481 486 525 523 478 528 534 503 464 520 507 464 481 504 535 488
499 509 519 591 586 558 536 486 509 430 432 388 428 408 378 399 410 420 416 417 447 494 423 427 441 422 421 371 398 364
399 382 410 413 440 444 408 404 421 473 446 434 416 433 430 398 425 444 445 446 490 447 446 469 448 421 439 404 408 412
502 472 483 469 507 531 504 465 460 406 411 438 441 411 452 407 418 427 437 403 481 505 461 469 432 418 436 433 417 403
412 404 427 400 430 413 414 422 426 445 378 416 384 420 410 392 393 416 455 392 461 469 427 452 438 459 454 473 474 438
450 495 487 485 473 455 487 502 525 499 489 473 486 505 521 517 469 491 523 550 590 558 496 541 524 555 508 528 540 535
551 557 557 522 500 542 569 529 564 521 536 604 533 570 519 529 545 547 546 537 580 516 488 544 515 507 521 474 510 515
464 450 433 449 487 467 424 426 424 423 426 418 433 412 441 406 437 412 393 449 418 410 460 363 377 403 419 394 395 390
378 391 403 403 367 398 418 388 409 385 379 366 376 368 370 382 422 392 370 352 337 377 381 360 331 348 366 320 329 316
301 321 349 329 314 336 320 333 308 311 317 305 314 313 345 321 328 323 293 330 286 309 292 314 278 275 317 306 316 316
293 323 319 327 316 299 322 326 315 321 307 315 323 355 355 323 337 329 350 326 344 336 357 350 333 364 363 361 353 306
355 357 343 326 339 345 341 327 378 352 342 366 334 397 340 375 351 340 364 375 347 350 342 331 384 399 375 343 349 352
337 315 335 329 336 340 341 349 307 374 362 342 351 385 329 355 361 376 360 355 355 342 365 350 358 337 336 297 338 342
352 366 365 310 322 296 336 333 349 340 329 329 338 334 331 315 329 314 322 280 317 284 312 330 292 297 313 268 295 331

269 305 314 268 291 280 309 283 288 258 285 288 306 283 291 282 284 273 307 303 300 322 262 268 272 296 275 264 268 286
288 277 270 296 280 266 279 291 293 288 277 284 283 261 284 272 269 267 274 299 277 271 273 270 279 243 256 278 258 309
257 270 275 247 315 269 282 255 283 272 242 268 279 238 297 251 250 233 262 271 277 256 265 302 261 275 264 258 260 275
243 259 266 259 289 284 291 262 274 253 268 265 252 314 267 265 243 277 251 264 266 259 248 277 243 238 256 255 261 250
253 272 275 245 245 279 237 209 248 257 257 261 239 279 261 256 252 237 271 310 274 240 253 256 272 245 231 292 255 251
269 253 257 219 226 258 237 237 247 222 239 240 251 247 218 209 216 250 239 222 223 228 217 199 231 216 236 245 253 246
227 214 230 241 222 229 244 212 243 212 205 243 241 210 218 241 231 237 224 240 206 227 208 233 220 204 242 226 234 225
209 222 233 233 210 210 228 232 261 214 208 224 195 213 233 237 214 236 186 250 236 232 203 199 240 185 202 225 208 239
209 215 219 194 208 217 217 198 196 229 225 243 208 215 215 222 223 229 219 217 197 229 230 197 246 188 215 241 239 209
211 224 234 219 203 213 215 229 242 232 234 250 218 229 248 229 223 226 199 237 230 208 208 252 278 214 232 210 220 206
227 229 197 216 203 206 219 198 224 218 223 200 203 213 220 191 210 202 180 204 187 192 211 216 214 188 180 219 191 229
161 149 217 224 190 201 218 201 206 203 211 210 223 201 217 203 194 196 211 231 202 205 189 199 195 196 210 178 204 215
212 199 184 223 222 184 208 199 215 192 192 208 200 206 198 197 209 229 212 202 219 218 167 233 190 192 233 204 210 204
186 200 202 212 215 205 204 191 189 210 218 197 233 209 211 216 219 232 190 236 213 237 188 214 211 210 198 189 194 227
227 220 234 183 190 212 170 228 185 226 238 197 205 246 202 211 236 201 205 216 192 218 233 242 220 218 198 198 199 224
218 211 209 208 260 206 214 231 215 190 210 231 230 216 245 214 228 229 226 227 229 214 247 226 238 239 253 261 250 234
245 234 278 262 252 266 245 225 244 243 247 216 212 188 237 215 259 216 243 195 200 217 227 236 231 244 219 216 232 255
218 262 217 224 223 176 202 201 209 223 220 231 197 226 219 219 225 228 199 225 170 189 204 211 195 188 218 219 185 187
190 183 195 173 190 192 189 209 181 218 190 199 196 191 220 184 173 205 181 210 227 199 203 204 200 233 182 151 201 217
187 176 186 195 201 219 194 214 197 202 208 212 194 206 195 189 196 215 229 189 205 206 200 187 208 178 197 206 201 200
196 196 192 192 191 229 220 169 173 219 215 196 199 196 219 176 210 188 160 199 195 184 190 185 197 170 211 173 211 198
172 170 187 187 211 227 188 198 201 229 214 236 228 218 216 231 184 192 204 190 208 199 199 216 215 196 214 215 215 175
195 188 227 214 208 222 200 204 214 196 209 212 236 222 210 164 196 188 186 195 197 188 183 208 184 226 214 222 184 214
228 205 200 206 181 192 219 232 224 214 202 210 212 188 224 177 204 204 224 194 185 220 206 196 195 212 194 202 201 206
187 176 189 171 186 197 206 188 183 187 170 199 190 177 204 191 186 181 180 181 181 163 177 159 175 197 188 194 191 202
172 181 170 187 204 158 181 164 171 188 172 177 159 165 192 183 180 151 194 181 172 178 172 129 174 169 190 150 192 163
180 164 162 173 172 183 175 183 165 149 189 150 191 184 171 165 172 180 173 147 196 165 170 135 165 164 176 197 159 185
180 178 182 173 180 161 164 180 175 171 156 189 185 155 197 178 186 183 168 170 192 162 160 173 148 155 167 159 181 141
164 167 158 146 153 188 146 160 173 157 171 162 156 150 157 172 171 172 175 165 163 159 185 161 169 167 157 163 137 148
160 164 168 175 154 157 151 185 156 168 161 171 181 157 172 191 170 144 169 146 168 187 160 160 171 165 144 149 162 172
167 181 180 162 150 194 141 160 161 190 154 164 169 142 175 162 174 154 159 148 159 150 143 151 145 157 190 181 147 169
149 153 167 175 154 156 189 169 180 169 157 164 175 176 162 190 145 167 184 167 182 186 171 181 172 146 151 164 176 192
172 176 200 163 161 197 181 170 174 182 154 183 175 145 167 154 170 182 172 155 170 173 171 161 159 127 148 160 160 145
155 186 154 159 156 159 139 175 163 157 150 155 158 147 149 163 141 139 134 137 159 150 148 157 168 151 134 122 146 140
160 154 150 161 155 161 148 149 151 157 143 136 134 151 165 139 154 171 153 143 159 155 153 155 158 170 143 150 165 154
147 155 135 150 155 141 186 145 144 127 137 126 143 154 133 139 151 160 161 161 145 143 181 147 150 159 143 176 157 173
175 148 148 158 156 155 141 161 148 164 144 143 122 145 149 157 156 142 142 163 149 151 144 161 147 153 181 156 153 158
155 148 139 162 139 134 132 148 151 149 129 160 153 159 141 148 151 156 145 157 154 180 131 137 153 157 148 178 157 145
149 157 160 153 150 150 140 127 174 173 149 169 151 170 141 137 148 139 168 167 169 143 129 136 160 148 163 148 165 154
147 147 142 142 144 173 140 149 157 150 171 169 131 131 162 160 161 145 149 156 159 131 127 161 114 148 141 134 129 132
127 155 137 132 144 168 145 129 150 156 156 144 144 137 147 116 131 132 145 132 135 148 134 139 135 130 131 139 163 132
145 133 134 171 129 140 149 154 120 136 133 143 153 156 135 141 139 161 149 133 143 142 149 130 159 162 155 135 157 144
128 142 170 135 134 142 158 130 143 150 139 151 135 137 157 132 143 133 130 157 158 149 120 144 137 155 141 121 140 142
167 134 117 132 155 132 137 175 126 122 133 132 135 146 153 148 151 147 122 132 132 145 131 143 132 158 159 132 158 113
122 122 130 147 113 134 129 123 123 130 143 158 130 126 148 144 105 143 109 146 139 121 142 128 139 121 115 140 134 105
129 156 130 134 149 133 109 141 148 130 144 127 153 140 121 139 126 148 125 132 130 151 136 137 157 143 150 117 142 153
108 142 128 134 136 135 102 136 123 157 132 128 136 144 141 109 133 111 142 156 146 139 131 137 133 123 125 134 137 129
153 155 120 131 131 146 161 158 147 153 144 122 132 129 120 150 123 127 150 142 142 116 136 141 147 122 112 149 129 137
143 132 120 132 128 105 163 130 128 114 113 108 125 144 120 113 125 112 135 143 116 131 125 139 112 130 135 118 135 119
116 127 144 135 131 128 108 131 121 117 129 127 121 129 150 112 125 134 119 117 126 122 117 109 133 146 113 106 107 126
128 117 105 126 131 111 132 127 137 135 136 106 92 108 127 106 114 120 149 141 111 120 153 116 121 112 130 130 119 123
123 156 116 135 122 115 127 117 104 118 103 146 131 145 112 130 132 147 116 107 131 118 113 130 141 115 122 107 133 113
117 121 128 116 103 108 123 135 120 126 125 133 112 111 127 113 115 103 130 117 109 112 109 98 112 126 103 112 101 130
125 130 122 130 116 127 117 104 127 126 112 93 115 107 131 91 109 105 114 98 99 120 111 129 112 113 111 117 104 120 121
108 119 133 114 122 112 120 109 117 111 112 132 109 114 114 122 120 131 150 149 130 109 117 121 122 106 123 136 140 150
118 103 114 119 102 114 116 121 136 106 112 121 130 131 123 131 115 133 114 109 115 107 107 121 109 143 118 111 143 116
119 149 98 111 122 119 129 142 122 139 115 122 115 108 104 118 127 98 130 108 107 108 125 108 105 120 117 123 115 116
118 125 114 123 107 129 119 111 142 99 118 105 99 112 116 121 108 122 117 99 127 121 109 122 105 132 125 130 126 117
123 116 119 106 113 109 109 95 130 102 109 113 114 129 122 117 119 103 111 120 144 128 95 132 99 122 113 112 108 117
116 93 131 95 90 100 93 144 127 129 123 111 112 140 115 130 112 103 106 116 107 111 104 94 107 105 103 101 108 107 102
115 121 130 120 105 114 95 116 104 100 117 102 98 126 99 112 105 98 104 104 115 108 122 97 106 119 118 106 111 98 109
108 113 102 117 125 106 107 127 89 109 108 100 99 104 97 91 98 95 111 121 102 113 93 135 123 107 109 121 118 103 133 99
102 98 126 103 136 114 102 122 123 102 114 108 115 106 125 122 123 120 113 109 108 118 116 97 117 98 104 109 95 104 109
101 111 132 107 104 112 109 108 112 103 127 105 99 118 111 113 101 104 125 93 131 134 87 105 112 95 107 88 98 107 103

104 106 86 102 84 99 122 121 97 104 102 106 107 97 105 108 89 102 109 95 116 99 98 92 125 105 81 121 105 106 89 119 105 108 118 117 120 101 90 116 98 97 92 86 95 98 132 106 106 103 120 107 109 102 105 97 100 92 103 117 103 116 93 108 99 102 108 101 92 88 125 102 105 107 104 104 120 118 112 123 101 78 105 91 93 94 95 118 105 118 102 99 88 94 87 107 99 114 119 100 122 107 113 105 108 103 93 104 103 100 87 112 107 116 108 106 115 116 97 99 87 108 105 111 127 95 114 90 98 108 99 114 111 98 102 87 84 103 103 100 111 104 104 84 82 117 98 125 92 115 102 84 111 99 87 102 89 95 98 106 132 106 106 105 102 95 95 105 86 111 94 109 93 86 118 95 109 116 90 113 113 90 109 101 84 107 113 108 99 98 99 120 109 119 97 89 111 104 81 108 113 104 92 82 105 104 121 113 98 104 103 106 101 98 112 95 135 115 80 109 100 93 100 106 89 108 101 108 95 76 108 111 111 77 95 108 97 109 111 119 112 103 100 101 107 104 93 103 91 105 84 105 98 114 97 99 104 93 98 90 89 105 113 101 118 78 85 99 109 78 97 98 108 118 102 86 92 113 98 103 108 113 102 106 106 88 108 94 92 104 103 98 102 98 94 109 86 108 121 109 106 97 103 95 103 77 106 101 95 93 106 90 117 90 98 107 104 98 113 99 104 85 84 101 81 94 87 102 106 94 87 100 87 90 101 103 108 103 106 92 114 90 87 99 87 103 80 93 82 100 116 114 105 103 93 103 107 100 98 76 91 97 95 101 91 92 104 98 85 106 109 94 90 101 106 99 104 100 92 113 116 90 98 85 84 79 103 111 82 105 95 117 106 107 93 89 94 92 103 90 99 92 84 111 93 107 97 93 105 95 87 97 108 90 104 108 105 93 93 92 80 85 81 97 95 99 94 99 103 106 99 113 86 108 91 102 99 104 88 104 128 101 105 103 93 92 100 82 84 80 86 93 92 93 100 79 91 87 86 79 88 92 100 81 82 94 88 102 80 87 107 102 97 79 89 91 101 86 102 90 79 99 86 80 94 94 112 104 99 98 99 97 80 74 90 95 98 91 94 99 85 85 79 111 98 89 95 99 87 95 86 109 85 100 90 97 115 114 113 118 109 91 79 91 95 100 84 90 90 94 99 111 109 92 102 99 97 101 90 89 74 71 79 94 88 82 112 90 90 82 74 92 84 101 101 101 94 93 93 87 102 81 98 88 92 92 92 98 80 93 81 95 97 82 101 112 111 88 98 102 98 88 99 90 95 86 104 95 89 90 94 86 84 106 84 93 103 95 79 103 90 97 84 85 78 99 99 109 76 98 102 103 106 104 99 91 89 93 104 95 92 109 106 92 102 93 103 76 100 92 82 102 101 89 102 86 87 86 94 78 77 95 86 87 87 90 89 92 105 85</intensities>

From the above chemical characteristics, elemental and spectral analysis the compound (VIIIa3) was assigned the structure as 2-tert-butylimino-6-amino-4-[2-isobutoxy-5(4-methyl-5-carboxy-1,3-thiazo-2-yl)]-phenyl-1,3,5-thiadiazine(VIIIa3)



(VIIIa3)

2-Tert-butylimino-6-amino-4-[-2-isobutoxy-5(-4-methyl-5-carboxy-1,3-thiazo-2-yl)]-phenyl-1,3,5-thiadiazine.

Similarly, 2-(3-thioamidoformami-dino-4-isobutoxyphenyl)-4-methyl-5-carbo-xy-1,3-thiazole (IIIa), were interacted with methylisocyanodichloride(VIIa),ethylisocyanodichloride(VIIb)p-cl-phenylisocyanodichloride (VIIe)o-tolylisocyano- dichloride (VIIf),m-tolylisocyanodichloride(VIIg),p-tolyliscyanodichloride(VIIh),by above mentioned method to isolate2-methylimino-6-amino-4-[2-isobutoxy-5(4-methyl-5-carbo -xy-1,3-thiazo-2-yl)]-phenyl-1,3,5-thiadiazine(VIIIa1), 2-ethylimino-6-amino-4-[2-isobutoxy-5(4-methyl-5-carboxy-1,3-thiazo-2-yl)]-phenyl-1,3,5-thiadiazine (VIIIa2), 2-p-chlorophenylimino-6-amino-4-[2-isobutoxy-5(4-methyl-5-carboxy-1,3-thia -zo-2-yl)]-phenyl-1,3,5-thiadiazine (VIIIa5), 2-o-tolylimino-6-amino-4-[2-isobutoxy-5(4-methyl-5-carboxy-1,3-thiazo-2-yl)]-phenyl-1,3,5-thiadiazine (VIIIa6), 2-m-tolylimino-6-amino-4-[2-isobutoxy-5(4-methyl-5-cabo -xy-1,3-thiazo-2-yl)]-phenyl-1,3,5-thiadiazine (VIIIa7) and 2-p-tolylimino-6-amino-4-[2-isobutoxy-5(4-methyl-5-carboxy-1,3-thiazo-2-yl)]-phenyl-1,3,5-thiadiazine(VIIIa8)respectively and enlisted in Table No. V-5.

Table No. V-5

	Compd. No.	2-Substituedimino 6-amino-4-[2-isobu -toxy-5(4-methyl-5-carboxy-1,3-thiazo-2-yl)]-phenyl-1,3,5-thiadizine	Yield %	M.P. ^{0}C
1.	(VIIIa1)	2-Methylimino-6-amino-4-[2-isobutoxy-5(4-methyl-5-carboxy-1,3-thiazo -2-yl)]-phenyl-1,3,5-thiadiazine	85	150
2.	(VIIIa2)	2-ethylimino-6-amino-4-[2-isobutoxy-5(4-methyl-5-carboxy-1,3-thiazo-2-yl)]-phenyl-1,3,5-thiadiazine	83	155
3.	(VIIIa3)	2-Tert-butylimino-6-amino-4-[2-isobutoxy-5(4-methyl-5-carboxy-1,3-thiazo-2-yl)]-phenyl-1,3,5-thiadiazine	84	166
4.	(VIIIa4)	2-Phenylimino-6-amino-4-[2-isobutoxy-5(4-methyl-5-carboxy-1,3-thiazo-2-yl)]-phenyl-1,3,5-thiadiazine	88	158
5.	(VIIIa5)	2-p-Chlorophenylimino-6-amino-4-[2-isobutoxy-5(4-methyl-5-carboxy-1,3-thiazo-2-yl)]-phenyl-1,3,5-thia-diazine	85	165
6.	(VIIIa6)	2-o-Tolylimino-6-amino-4-[2-isobutoxy-5(4-methyl-5-carboxy-1,3-thiazo-2-yl)]-phenyl-1,3,5-thiadiazine	82	160
7.	(VIIIa7)	2-m-Tolylimino-6-amino-4-[2-isobutoxy-5(4-methyl-5-carboxy-1,3-thiazo-2-yl)]-phenyl-1,3,5-thiadiazine	82	162
8.	(VIIIa8)	2-p-Tolylimino-6-amino-4-[2-isobutoxy-5(4-methyl-5-carboxy-1,3-thiazo-2-yl)]-phenyl-1,3,5-thiadiazine	80	176

References

1. Li C.J. and Chan T.H., Tetrahedron, 55, 1999, 11149.
2. Cave G.W.V., Raston C.L. and Scott L., Chem.Commun., 2001, 2159.
3. Imrie C., Kleyi P., Nyamori V.O., Gerber sI.A., Levendis D.C. and Look J., Journal of Organomet. Chem., 692, 2007, 3443.
4. Anastas P.T. and Warner J.C., Green Chemistry, Theory and Practice, Oxford University Press, New York, 1988.
5. Nassar Ekhalass, Journal of American Science, 6(8), 2010.
6. Abdel-Aziz H.A., Saleh T.S., El-Zahabi H.S.A., Arch. Pharm., 343(1), 2010, 24-30.
7. Toyata K. Shinkai H. Etou H. Kamimura A. Eguchi C. Oosumi K., Turuo T., Eur. Pat. EP 330, 1989,470 (cl. C07D211/90), Chem. Abstract., 112, 1990, 158059.

8. Wang G.T., Wang X., Wang W., Hasvold L.A., Sullivan G., Hutchins C.W., O'Conner S., Gentiles R., Sowin T., Cohen J., Gu W.Z., Zhang H., Rasenberg S.H., Sham H.L., Bioorg. Med. Chem. Lett., 15(1), 2005, 153-158.
9. Baldwin J.J., Engelhardt E.J., Hirschmann R., Ponticello G.S., Atkinson J.G., Wasson B.K., Sweet C.S., Scriabine A., J. Chem., 23, 1980, 65-70.
10. Jakhar A. and Makrand J.K., J.Chem.Res., 4(3), 2010 , 238-240.
11. Braghiroli D., Puja G., G.Cannazza, Tait A., Parantai C., Losi G. and Baraldi M., J.Med.Chem., 45(12), 2002 2355-2357.
12. Ei Bialy S.E., Abdelal A.M., Shorbagi A.N., Kheria., Pharma.Med.Chem., 338, 2005, 38-43.
13. Witvrouw M., Arranz M.E., Panneciuque C., Declercq R., Jonckheere H., Schmit J.C.,Vandamme A.M., Antimicrob.Agents Chemother., 42, 1998, 618-623.
14. Liu X., Yan R., Chen N., Xu W., Molina M.T. and Vega S. Molecules, 11(11), 2006, 827-836.
15. Blum R.H. and Carter S. K., Ann.Inter Med., 80, 1974, 249-259.
16. Wan Z.Y., Shi H.X. and Shi H.J., J.Heterocyclic Chem., 38, 2001, 335.
17. Zhang L.X., ZhangA.J., Hu M.L. and Lei X.X., Acta Chim.Sinica, 61(6), 2003, 917.
18. Zhang Y.,Qiao R.Z. and Zhang Z.Y., J.Chin.Chem.Soc., 49(3), 2002, 369.
19. Ertan M., Bilgin A.A., Palaska E., Yulug N.and Arznei., Forsch/Drug Res., 42(1), 1992,160.
20. Lin T.S., Zhu L.Y., Xu S.P., Divo A.A. and Sartorelli A.C., J.Med.Chem., 34, 1991, 1634-1639.
21. Huang Z.H., Chen Y.N., Menon K., Teicher B.A., J.Med.Chem., 36, 1993, 1797-1801.
22. Bayoumi Y.A. and Hafez Y.M., Acta. Biologica Szegediensis, 50(3-4), 2006, 131-136.
23. Muelas S., Mario A. and Cerecetto H., FOLIA PARASITOLOGICA, 48, 2002, 105-108.
24. Hu G.Q., Xie S.Q., Huang W.L.and Zhang H.B., Chin.Chem.Lett., 16(6), 2005, 723-726.
25. Scendo M., Poddebnick D. And Malyszko J., J.Appl.Electrochem., 33, 2003, 2337.
26. Dafali A.,Hammouti B.,Touzani R., S.Kertit S.,Ramdani A.and Kacemi K. Anti-Corros.Meth.Mater., 49, 2002, 96.
27.Tayade D.T., A Contribution to the chemistry of nitrogen, nitrogen and sulphur containing heteroacyclic and heterocyclic compounds, Ph.D. Thesis, Amravati University, Amravati, 1996.
28. Shelke M.S., Synthesis of 1,3-Diformami dinothiocarbamide, hydrochloride and their cyclisaton to substituted imino/amino-1,3,5-thiadiazine hydrochlorides and 1,3,5-triazine, Ph.D. Thesis, S.G.B. Amravati University, Amravati 2005.
29. Panpalia R.C., Studies in the chemistry of some new thiocarbamides and Hector's base, Ph.D. Thesis, S.G.B. Amravati University, Amravati, 2006.
30. Tayade D.T. and Chincholkar M.M., Acta Ciencia Indica, XXI (i), 1995, 37-38.
31. Pathe P.P., Ambekar M.W., Mimdeokar N.M. and Paranjpe M.G., Ind. Jr. Chem., 59, 1982, 670.
32. Deohate P.P., 'Application of N-phenylisocyanodichloride, N-Phenyl-S-chloroisothiocarbamoyl chloride and iodine in the synthesis of heterocyclic system', Ph.D. Thesis, SGB, Amravati University, Amravati, 2004.
33. Hector D.S., Ber., 22, 1889, 1176.
34. Hector D.S., Ber., 25, 1892, 779.
35. Hector D.S., Oefuers Kong Vet, Akad., 89, 1892.
36. Ghosh S.K. S.K.,Advanced Organic Chemistry, 2nd Ed., Calcatta, 1998 (a) P-410, (b) P-412.
37. Lapman G., Pavia D. and Kriz G., Introduction to Spectroscopy, Asia a Pte Ltd., 3rd Ed., Singapoor, 2004, (a) P-68-69, (b) P-43.
38. Tayade D.T., Asian Jr. of Chemistry, 7(4), 1995, 890-91.